

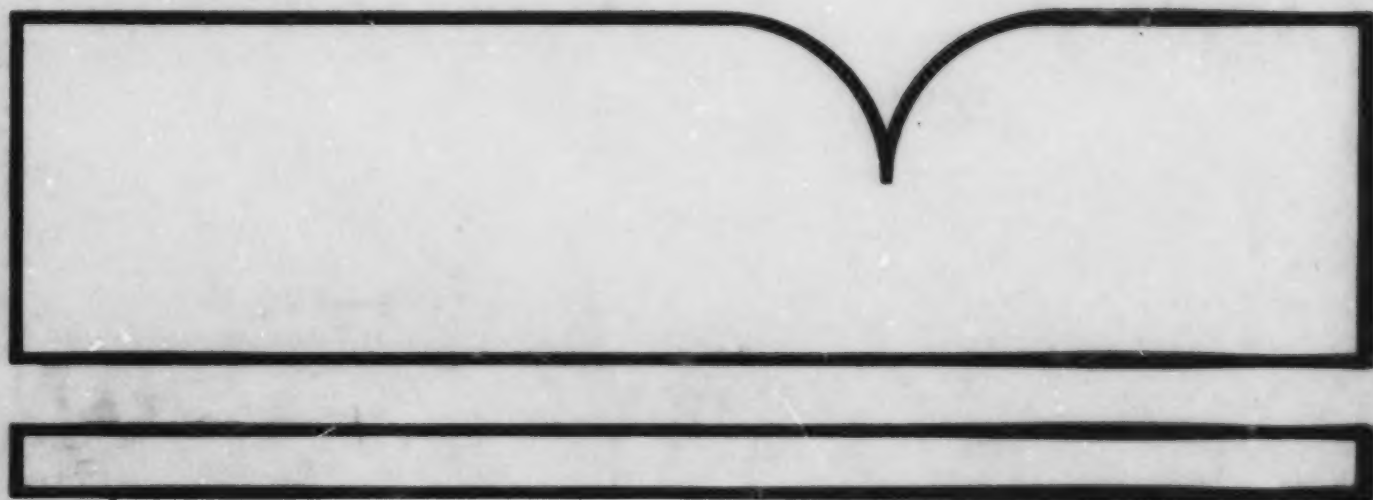
Safe Disposal Methods for Agricultural
Pesticide Wastes

Iowa State Univ.
Ames

Prepared for

Municipal Environmental Research Lab.
Cincinnati, OH

May 81



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SAFE DISPOSAL METHODS FOR AGRICULTURAL PESTICIDE WASTES

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FOREWORD

The Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. The complexity of that environment and the interplay between its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution, and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems for the prevention, treatment, and management of wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, for the preservation and treatment of public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research: a most vital communication link between the researcher and the user community.

This study describes a systemic evaluation of disposal systems for control of Agricultural Pesticides Wastes. The evaluation included detailed chemical studies of the disposal systems and their surrounding environments, identification and counts of bacterial populations, entomological studies, estimation of pesticide volatilization rates and convenience of systems operation.

Francis T. Mayo
Director
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Laboratory

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ABSTRACT

A systematic evaluation of disposal systems for diluted waste pesticides was conducted at two Iowa State University experimental farms. One system, located at the Horticultural Research Station, consisted of a 30,000-liter concrete-lined pit filled with a layer of soil between two layers of gravel and equipped with a rain-activated cover. The second system, located at the Agronomy-Agricultural Engineering Center, involved two polyethylene-lined pits. One pit had been in use for 14 years and was uncovered. The other pit was constructed in 1978 and was covered.

In addition to investigation of these macropit disposal systems, a study was made of 56 covered, vented minipits where additions of pesticides were controlled as opposed to the macropits where additions were dictated by the operational needs of the experimental farms.

Evaluation of the pit disposal systems included detailed chemical sampling of the systems and their surrounding environments, identification and counts of bacterial populations, entomological studies, estimation of pesticide volatilization rates, and evaluation of pit design for efficiency, effectiveness, and convenience of operation.

The covered, concrete-lined pit was found to be a safe, effective disposal method for chemical wastes if properly managed. After nine years of use and deposition of more than 40 different chemicals, the disposal pit continues to be biologically effective, and the chemical degradation is occurring. No leakage or contamination of the surrounding environment has occurred.

The polyethylene-lined pits were not found to be completely satisfactory systems for pesticide waste disposal. The older open pit that had been in use for 14 years did not contain chemical wastes and did adversely affect the surrounding area. The newer polyethylene-lined, covered pit apparently leaked during the first year. Although leakage was not massive, complete containment of the chemical wastes was questionable, suggesting that this type of pit would be unsatisfactory, especially in areas where ground water contamination is likely to occur.

This report was submitted in fulfillment of Grant No. R804533 by the Iowa State University under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period November 1, 1976 to October 31, 1979.

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- 93 Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
No aeration, no nutrients, and no other pesticides. 121
- 94 Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
~1 l/min air, no nutrient, and no other pesticides. 122
- 95 Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
No aeration, 0.1% peptone nutrient, and no other pesticides 123
- 96 Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
~1 l/min air, 0.1% peptone nutrient, and no other pesticides 124
- 97 Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
No aeration, no nutrient, and 5 other pesticides. 125

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ACKNOWLEDGMENTS

Personnel from 5 departments and the Energy and Mineral Resources Research Institute participated in this study. Participants listed as authors were project leaders and those primarily responsible for preparing the final report.

Lynette Johnson and Dennis Jones from Agricultural Engineering participated in the Volitization study and did design features of proposed new pit modifications, respectively.

John Pesek was facility coordinator for Agronomy and Ray Nicholson is station superintendent.

Paul Hartman was major professor for Layne Johnson who did the microbiological survey of the Horticulture Station disposal pit. Michael A. Grant was research assistant involved in other microbiological studies directed by Fred Williams.

Dave Staniforth, herbicide specialist, from the Botany Department served as consultant to project leaders.

John Richards, Associate Chemist, from the Energy and Mineral Resources Research Institute had major responsibility for collection of samples from disposal pits, water sources and the analytical procedures for all pesticides. He also participated in preparation of reports.

Al Kemp, station superintendent at the Horticultural Station, was responsible for coordination of pesticide use and disposal.

All of those listed served essential roles in this complex study and deserve recognition for their contribution. Especially John Richards of EMRRI and Dennis Jones were major contributors.

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SECTION 1

INTRODUCTION

Concern has grown in recent years over pollution of water supplies, soils, and the overall environment by improper disposal of waste pesticides and other toxic chemicals. Few attempts had been made to develop safe disposal methods and provide recommendations to private users or commercial applicators. Regulatory agencies, however, have been receiving continuously greater pressure to regulate or require safe disposal.

As a result, the agricultural and horticultural research farms at Iowa State University deemed it necessary to provide safe disposal for a wide variety of diluted waste pesticides. A polyethylene-lined disposal pit was developed at the Agronomy-Agricultural Engineering Center in the early 1960's. The pit was not covered and served only as a dump site for sprayers. In 1969-70, when the new Horticultural Research Station was established, a complete pesticide-handling facility was established for storage, mixing, and waste disposal. Neither of these systems had ever been evaluated for effectiveness or environmental safety, however.

A systematic evaluation of both disposal systems was initiated to determine their effectiveness in terms of containment of waste pesticides and chemical degradation, and to seek improvements for other users. This evaluation involved chemical analytical studies to determine degradation rates for individual pesticides, entomological bioassays to confirm the analytical findings, bacteriological studies to determine microbial characteristics and survivability, and volatilization studies to estimate volatilization rates of selected pesticides and predict the evaporation rate of water. In addition, a complex system of minipits was established to study selected individual pesticides and combinations with respect to the influence of aeration and supplementary carbon (peptone) on bacterial activities and degradation rates.

Each system has been characterized as to its effectiveness and its relationship to environmental safety. Design and operational suggestions were developed for generators of dilute pesticide wastes.

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SECTION 2

CONCLUSIONS

- 1) A reinforced concrete pit (3.7 m wide by 9.1 m long with an average depth of 1 m) located at the Iowa State University Horticultural Research Station and equipped with a rain-activated mobile cover was found to be a highly satisfactory disposal system. The pit was filled with two 30-cm layers of gravel with an equal layer of soil between.

After ten years of use for waste pesticide disposal, the pit has not contaminated the surrounding environment, and biological activity within the system is essentially normal. The system is fully effective to date.

Atmospheric pollution as a result of evaporation of dilute liquid pesticide was negligible.

- 2) An uncovered pit lined with film plastic in use at the Iowa State University Agronomy-Agricultural Engineering Station for many years has not been effective. Residues have not been fully contained, and the immediately surrounding area has been affected.
- 3) A newly constructed, covered polyethylene-lined pit at the Agronomy-Agricultural Engineering Station was partially effective the first year. The water level did fluctuate, however, which indicates some leakage. The design and construction procedures were adequate, but the liner was not satisfactory in preventing leakage.
- 4) Geographic, environmental, and soil conditions influence the design and acceptability of materials.
- 5) Similarly used pits can be chemically sampled for contents, leakage, and environmental safety. There are limitations on the accuracy of estimating residual contents, but leakages can be detected.
- 6) Bacterial activity continues in the concrete pit after dumping more than 45 kinds of pesticide wastes. The soil layer contains greater than 3% organic matter, which continues to promote a high level of aerobic activity.
- 7) Replicated studies of selected single pesticides and combinations thereof in plastic garbage cans partially filled with soil (minipits) provided accurate decomposition data and no evidence of harmful effects on bacterial populations.

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- 8) Controlled laboratory studies of pesticide volatilization in saturated and unsaturated vapor conditions indicate that pesticide concentrations in the air were not significantly affected by whether or not water was being evaporated.
- 9) Models were developed for predicting pit water evaporation rates from climatological data. Pit surface area required to accomodate a particular volume of daily disposals can be determined from estimation of evaporation rate.
- 10) Size and system cost vary for specific localities, depending on location, soil-water relationships, economic condition, population density, and user needs.
- 11) A system for washing, crushing, and disposing of 5-gallon containers was incorporated into the total system at the Horticultural Research Station

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SECTION 3

DESCRIPTION OF SYSTEMS EVALUATED

MINIPITS

To test the pit method of disposal of excess pesticides under conditions of controlled pesticide additions (amounts and timing, as opposed to additions to the macropits as dictated by the experimental farms' operational needs), a minipit system was designed and installed at the Agronomy-Agricultural Engineering Research Center. The minipits were small enough to allow mixing of the entire pit for sampling, yet large enough that the amount removed was inconsequential. The small pits were also a check on the assumption that pit size was not a significant factor in the dissipation of pit-disposed pesticides. Although additions to the minipits were controlled, the physical environment for the minipits was not; temperature, humidity, wind speed, etc. were essentially the same as for the macropits, as all pits were exposed to outside weather conditions.

The minipits themselves were about 115-liter containers (Sears 30-gallon polyethylene garbage containers, cat. no. 11X 6375N2), which were installed in a 7 x 8 grid as shown in the schematic in Figure 1. The containers were partially buried (~45 cm deep) in an area that had water and electrical power available. Before addition of pesticides, 15 kg of soil (on a dry weight basis, but the soil was not dried) was weighted into each minipit. The soil, a sandy-loam, was from an area of the Horticulture Research Station that was not known to have had pesticides applied to it. The soil had 2 to 3% organic matter and a mechanical analysis of 65% sand ($>62\mu$), 25% silt ($62-2\mu$), and 10% clay ($<2\mu$). About 55 liters of water were also added before addition of pesticides. After addition of pesticide, the final volume of the soil-water-pesticide mixture was brought to a depth equal to 65.7 liters (60 liters of liquid; 5.7 liters of soil, assuming a soil density of 2.65 g/ml).

The six pesticides studied (four herbicides and two insecticides) were chosen on the basis of three factors. First, they represented six different families of pesticides. The four herbicides--alachlor, atrazine, trifluralin, and 2,4-D--represent the acetanilides, triazines, dinitroanilines, and acid herbicides, respectively. The two insecticides--carbaryl and parathion--represent the carbamate and organophosphorus insecticides, respectively. Second, the pesticides studied were chosen on the basis of current and projected use in Iowa (Jennings and Stockdale, 1978) and the Midwest; therefore, no chlorinated hydrocarbon insecticides were chosen. And third, the pesticides chosen were ones for which the analytical methodology was either already established by the researchers involved or easily adapted.

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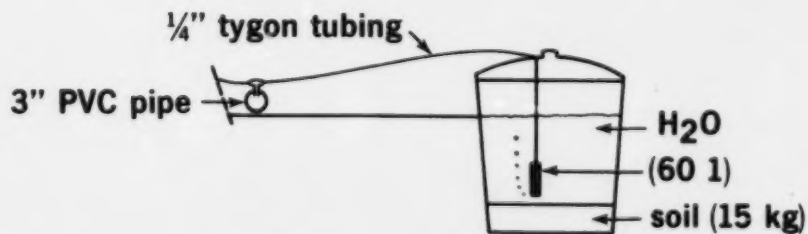
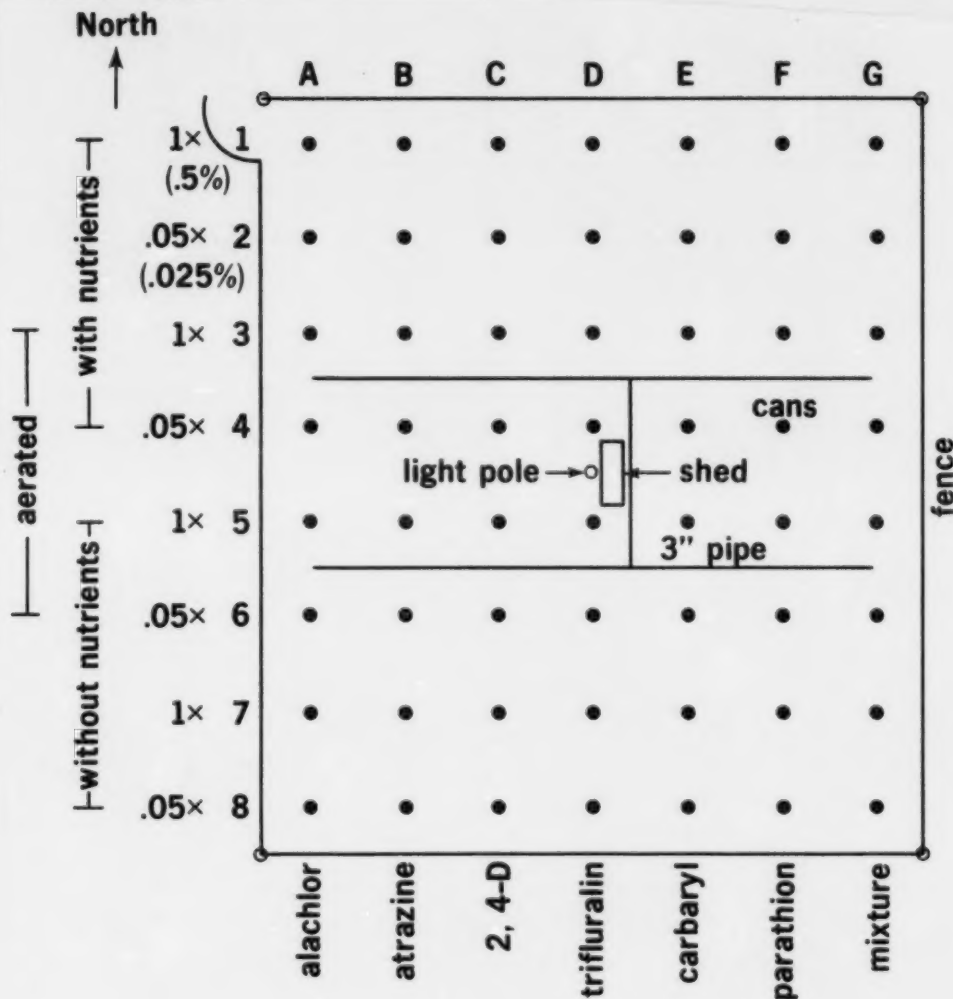


Figure 1. Schematic diagram of minipit area.

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As shown in the schematic (Figure 1), these six pesticides were studied individually and as a mixture of all six. There were two concentration levels of 0.5% (1X) and 0.025% (.05X) of active pesticide ingredient (w/v, based on 60 liters of liquid, or 300 and 15 grams, respectively). The 1X level of 0.5% approximates the maximum concentration of a waste pesticide disposal (i.e., the concentration of a solution or suspension actually used in application). The .05X level of 0.025%, a factor of 20 dilution, approximates the concentration that might be experienced during rinsing or washing application equipment and "empty" pesticide containers. Minipits in east-west row numbers 1, 3, 5, and 7 contained the high concentrations, and minipits in row numbers 2, 4, 6, and 8, the low concentrations.

To determine if added nutrients or aeration enhanced dissipation of the pesticides added to the minipits, one-half of the minipits, row numbers 1 through 4, received an addition of a nutrient source for microorganisms. Peptone was used and was added at the rate of 0.1% (w/v, or 60 grams). The other one-half of the minipits, row numbers 5 through 8, received no added nutrients. One-half of the minipits, row numbers 3 through 6, were aerated at the rate of about 1 liter/min through the manifold system shown (at about 35 KPa), with fritted glass dispersion tubes at the bottom of the minipits used as bubblers. Minipits in row numbers 1, 2, 7 and 8 had only naturally occurring aeration. All the minipits were covered with the garbage can lids (except at sampling) to exclude precipitation. The covers were vented by drilling four 0.6 cm holes in the cover handles.

On 4/27/77 the pesticides were added to the soil (15 kg) and water (~55 l) in each minipit. After bringing the total volume to the desired 65.7 liters (using a point-gauge to determine the desired depth), the soil-water-pesticide mixture was vigorously stirred using a propeller stirrer. The drive for the stirrer was a 1500 rpm electric drill; the stirrer and point-gauge are shown in Figure 2. Sampling, as shown in Figure 3, was performed by slowly lowering and raising a 125 ml sample bottle through the mixture while stirring continued. The top hole of a two-hole stopper in the mouth of the bottle allowed air to escape as sample flowed through the bottom hole. This allowed the sample to be taken over a period of several seconds and thus a depth-integrated sample to be obtained in case stratification of the solid-liquid mixture was occurring.

In addition to sampling performed the day of pesticide additions, samples were taken periodically thereafter (after first adding water to reestablish the 60 liter liquid volume) at increasing time intervals. The dates of sampling (and readdition of pesticides to certain pits from which the pesticides had dissipated) and results of sampling and analyses are given in the sections on chemical studies.

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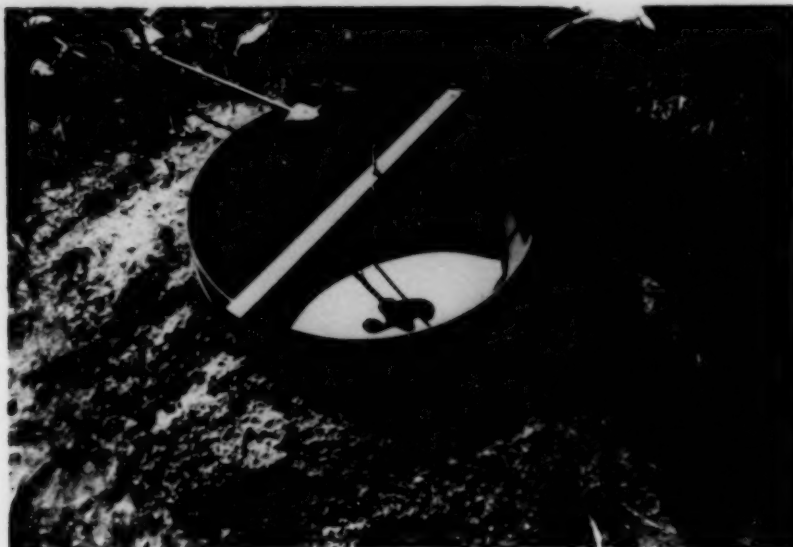
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9-1/8 Figure 2. Minipit point gauge and stirrer.



Figure 3. Stirring and sampling minipits.

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The macropit at the Agronomy-Agricultural Engineering Research Center (AAERC) was constructed in 1977 and used for the 1978 and 1979 seasons (one disposal was made in the fall of 1977). As shown in Figures 4 and 5, the macropit was a dug pit 1.5 m deep (1.7 m deep from the rock surface) and with surface dimensions of 7.3 x 13.7 m for an area of 100 m² (however, the rock surface has the dimensions of 7.9 x 14.3 m for 113 m²). The total volume of the pit is 74 m³ (90 m³ to the rock surface).

Also shown are five vertical tile that were used for liquid sampling and depth to the liquid surface measurements, and a disposal distribution system. This system was a galvanized metal pipe in an H shape buried within the rock. It was run to the surface with a hose and connector that could be coupled with outlets on sprayer rigs. Waste liquid pesticide mixtures were then distributed over the pit area by gravity flow and drainage from the holes drilled in the pipe. Records of the volumes dumped, the names of pesticides contained in the liquid, their concentrations on an active ingredient basis, and the dates of the disposals were kept by the research center personnel using the pit. The wooden canopy constructed over the pit with open sides was intended to keep precipitation out of the pit. The 3.6 m clearance allowed application equipment to drive through; the rock supported their weight.

As shown in Figure 6, the pit was lined with two sheets of continuous black polyethylene sheet encompassed on both sides by layers of sand. Metallic hardware cloth was also laid on the outside soil-sand interface to stop burrowing rodents. The pit was backfilled to a depth of 1.22 m with the original soil, and then a 0.46 m layer of crushed rock was added to the surface.

HORTICULTURAL RESEARCH STATION*

The original macropit system was constructed in 1969-70 and has been in continuous use since that time.

Beginning with this study in 1977, four micropits were placed along the west end of the macropit to collect equivalent volume samples of each lot of pesticide waste dumped into the macropit. The four micropits were designated check, fruit, vegetable, and ornamental to correspond with pesticide uses at the station. Details of location and micropit profile are shown in Figures 7 and 8. Since only an incomplete history of disposal was available for the macropit, it was believed that chemical analysis of the content would be more accurate where the exact history was known.

* Architectural drawings of the AAERC and Horticultural Research Station pits are in feet and inches; 1 ft. = 0.3048 m, 1 in. = 2.54 cm.

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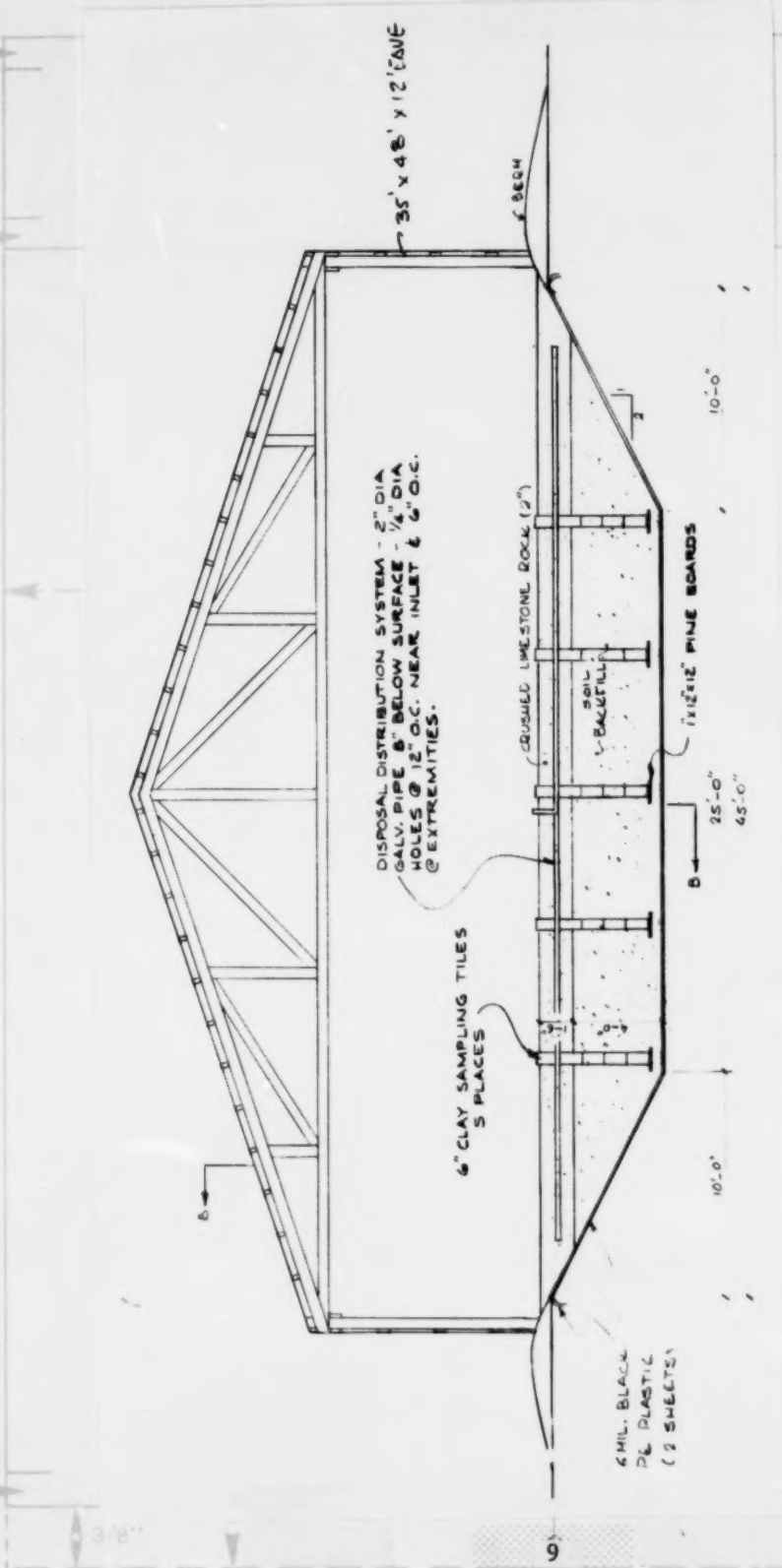


Figure 4. Cross-section view of AERC macropit.

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Figure 5. Overhead view of AAERC macropit.

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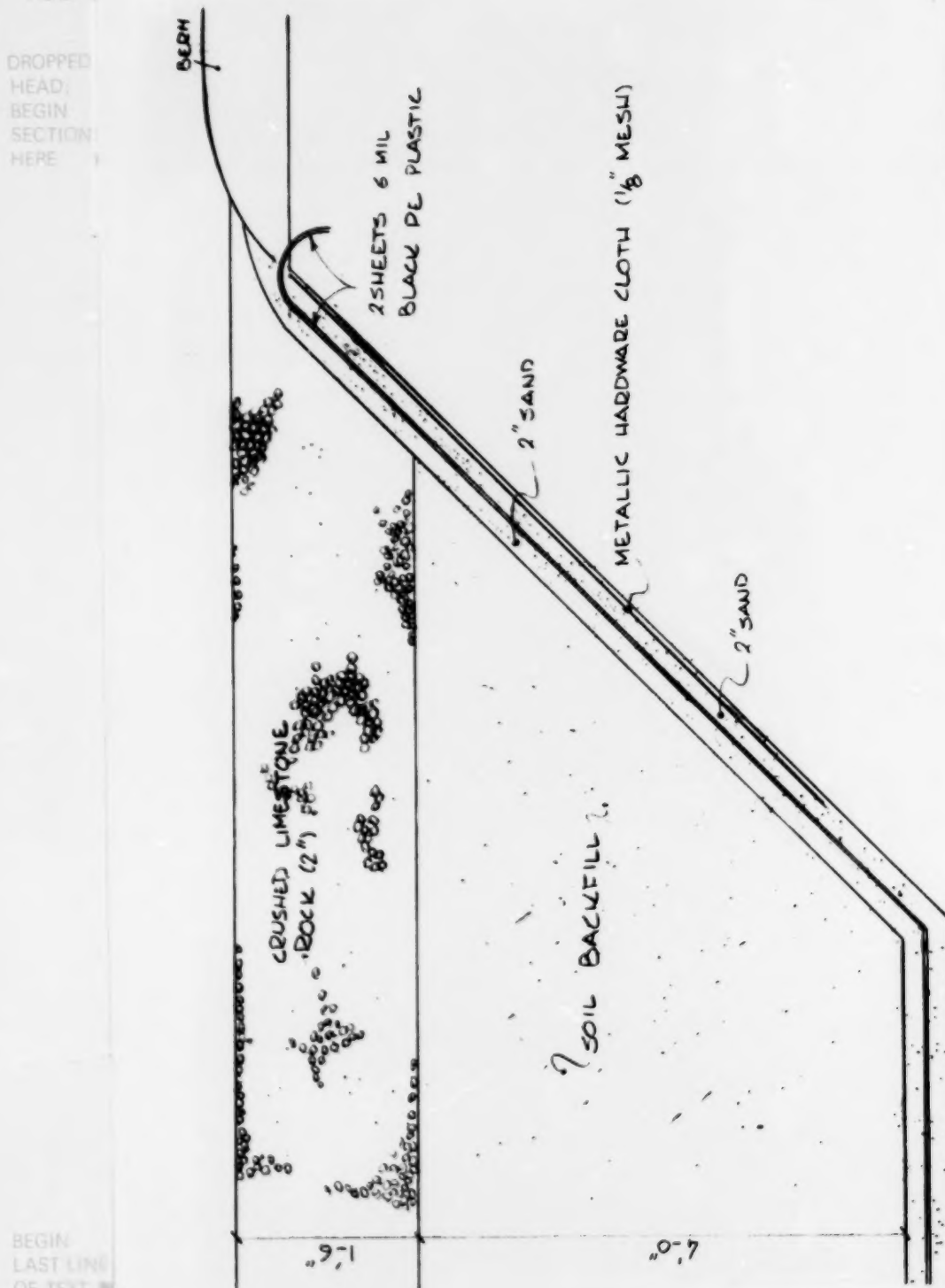
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Figure 6. Detailed view of AAERC macropit wall construction.

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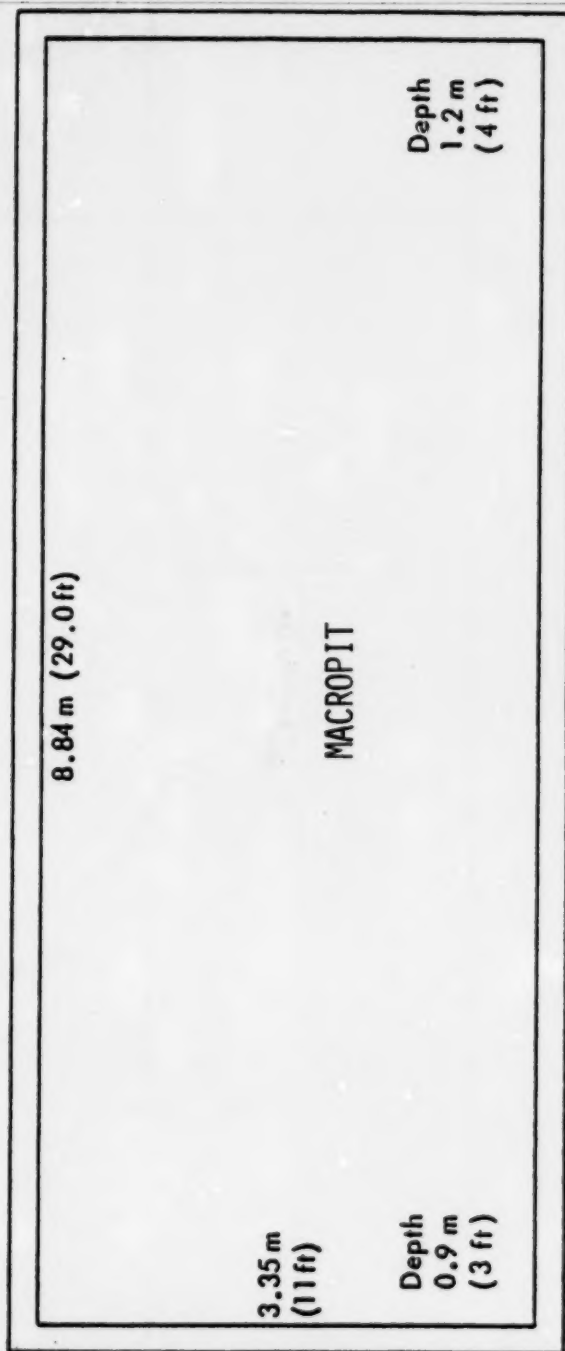
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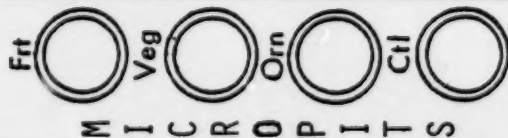
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TOP VIEW



Frt - Fruit
Veg - Vegetable
Orn - Ornamental
Ctl - Control

Figure 7. Iowa State University Horticultural Research Station Disposal Pits.

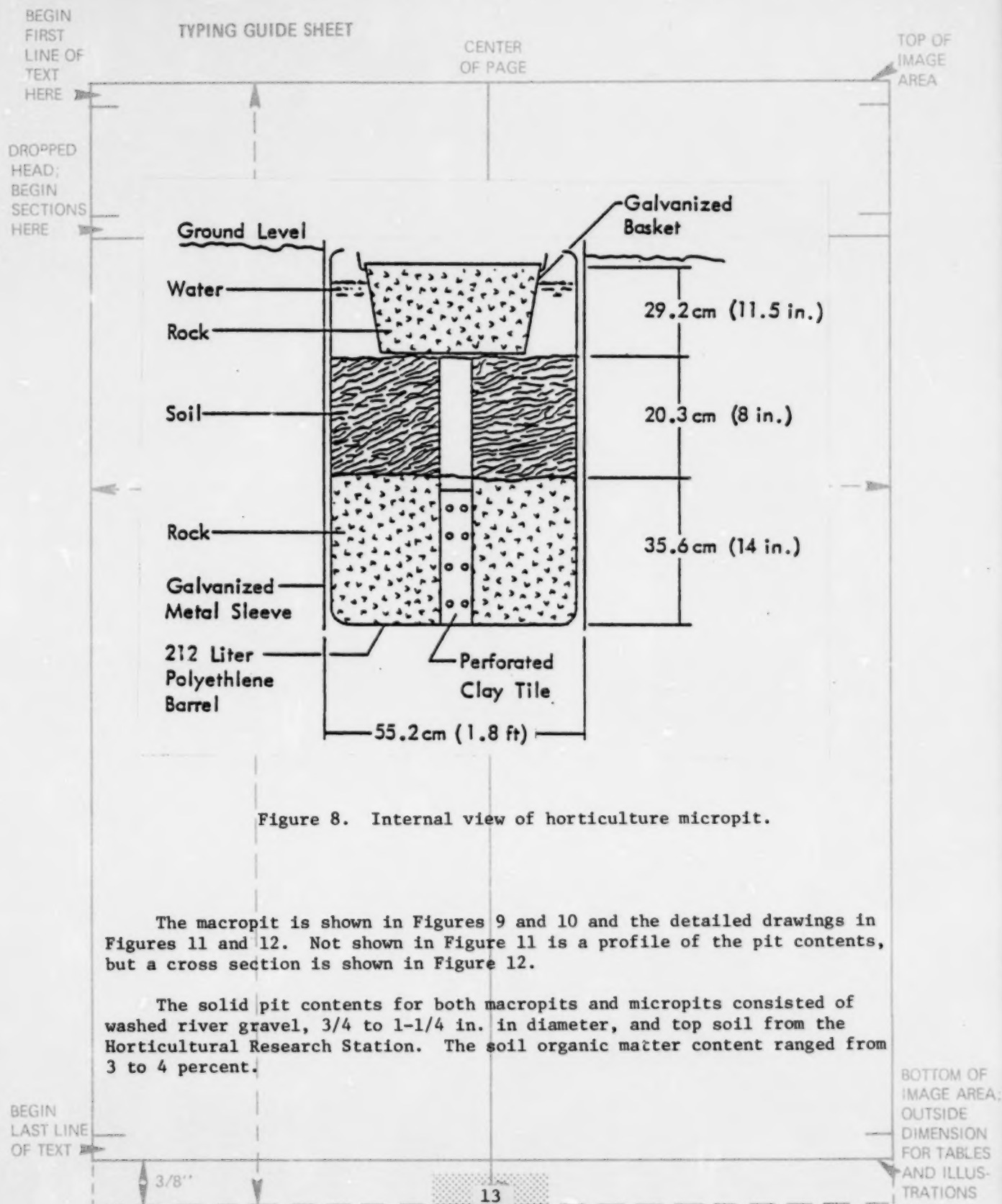
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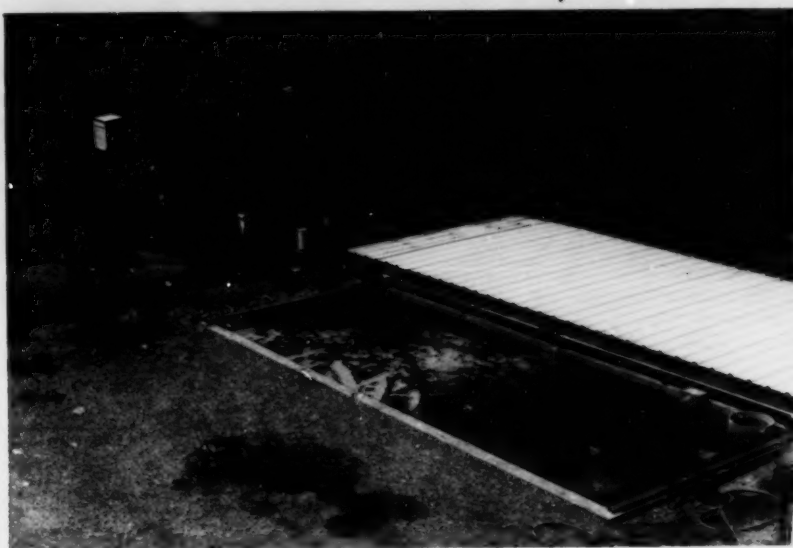


Figure 9. Aerial view of Horticultural Research Station macropit with cover open and climatological data recording equipment.

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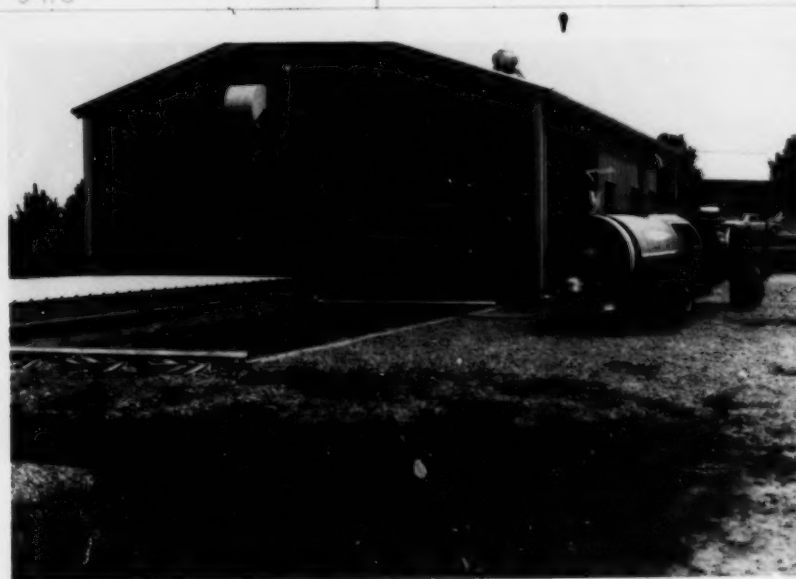


Figure 10. Horticultural Research Station macropit as related to the pesticide storage and handling building.

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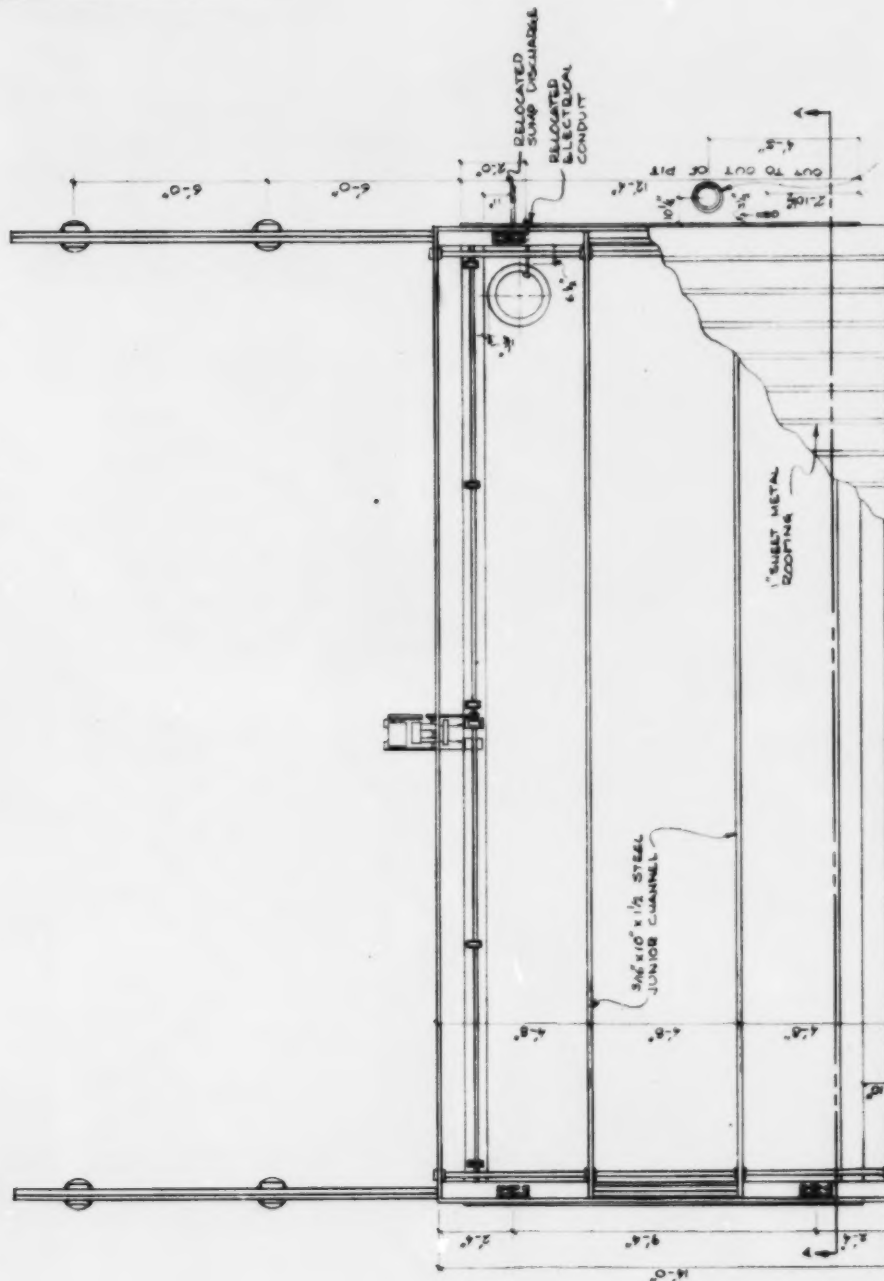


Figure 11. Horticultural research station macro disposal pit: Top View

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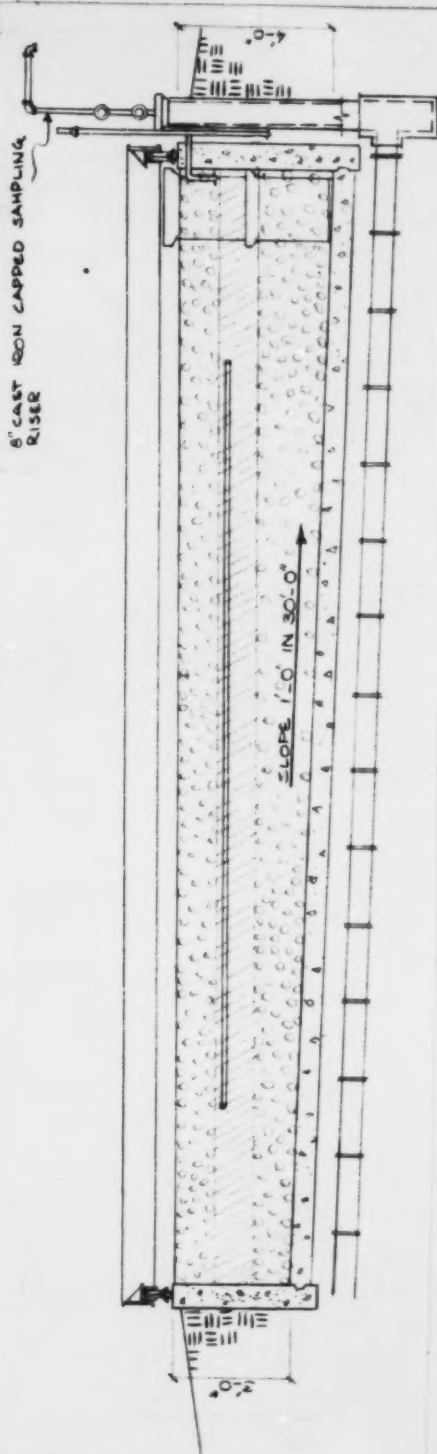
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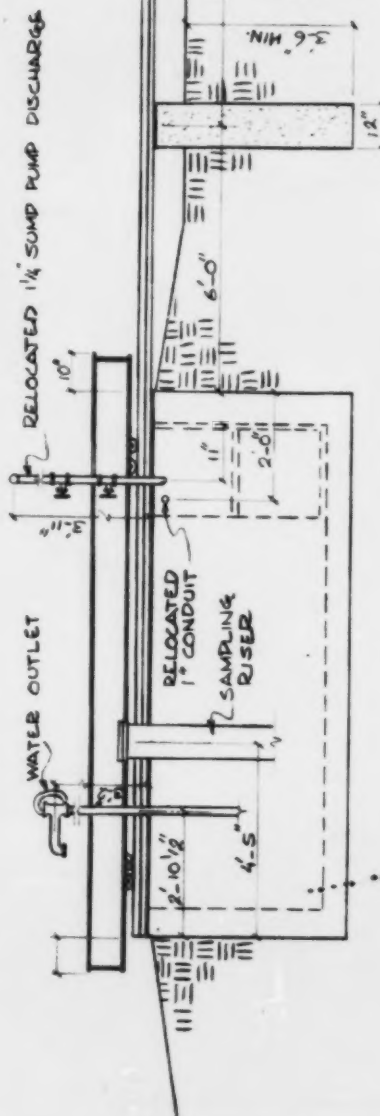
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Figure 12. Horticultural research station macro disposal pit: a) vertical section; b) end elevation.

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SECTION 4

CHEMICAL STUDIES

INTRODUCTION

The major inputs to the assessment of the utility of the various pesticide disposal pit systems discussed in this report have been the chemical analyses for pesticide residues and decay products. In this section the experimental procedures are described and the results are discussed for the analyses of the following pit systems:

- one (1) Horticulture macropit;
- four (4) Horticulture micropits;
- two (2) Agronomy macropits; and
- forty (40) Agronomy minipits.

Analytical procedures for the measurement of two insecticides in an additional sixteen (16) Agronomy minipits are described in Section 6, Entomological Studies. Experimental procedures are also described here for the analyses of soil, water and air in regions adjacent to and remote from the various pit systems under investigation.

The results of all these chemical analyses are presented in tabular and graphical form and discussed relative to the acceptability of the different disposal systems and the best methods for monitoring of the pesticide residues and their degradation products.

For ease in identification of pesticides discussed in this section, an alphabetical listing with cross references, chemical names and structures is given in Table 1.

EXPERIMENTAL

Sample Collection and Preparation

Samples were collected from four different disposal pit systems, Horticulture macropit, Horticulture micropits, Agronomy macropits and Agronomy minipits. These were collected and prepared as described below.

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TABLE 1. ALPHABETICAL LISTING OF PESTICIDES DEPOSITED AT DISPOSAL SITES
(HORTICULTURE, AGRONOMY #2 AND MINI PITS) WITH CHEMICAL NAMES AND STRUCTURES

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Pesticide	Chemical Name	Structure*
Aatrex	See Atrazine	
Alachlor	2-Chloro-2',6'-diethyl-N= (Methoxymethyl)-acetanilide	See A-1
Alanap	See Naptalam	
Amex	See Butralin	
Amiben	See Chloramben	
Atrazine	2-Chloro-4-(ethylamino)-6= (isopropylamino)-1,3,5-triazine	See A-2
Azinphos-methyl	0,0-Dimethylphosphorodithioate S-ester with 3-(mercaptomethyl)= 1,2,3-benzotriazin-4(3H)-one	See A-3
Banvel D	See Dicamba	
Basagran	See Bentazon	
Benlate	See Benomyl	
Benomyl	Methyl-1-(butylcarbamoyle)= benzimidazol-2-ylcarbamate	See B-1
Bensulide	0,0-Diisopropyl phosphorodi- thioate S-ester with N-(2= mercaptoethyl)-benzenesul- fonamide	See B-2
Bentazon	3-Isopropyl-1H-benzo-2,1,3= thiadiazin-4-one-2,2-dioxide	See B-3
Bexton	Unknown	
Bladex	See Cyanazine	
Bravo	See Chlorothalonil	
Buthidazole	1-(5-Butyl-1,3,4-thiadazol-2= yl)-3-methyl-5-hydroxyl-2-imida- zolidinone	See B-4
Butoxone	See 2,4-DB	
Butralin	4-(1,1-Dimethylethyl)-N-(1= methyl propyl)-2,6-dinitro- benzeneamine	See B-5
Butylate	S-Ethyl-N,N-diisobutylthiocar- bamate	See B-6

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Pesticide	Chemical Name	Structure*
Captan	N-(Trichloromethylthio)-3a,4,7,- 7a-tetrahydrophthalimide	See C-1
Carbaryl	1-Naphthyl-N-methylcarbamate	See C-2
Casoron	See Dichlobenil	
Chloramben	3-Amino-2,5-dichlorobenzoic acid	See C-3
Chlordane	1,2,4,5,6,7,8,8-Octachloro-2,3,- 3a,4,7,7a-hexahydro-4,7-methano- indene	See C-4
Chlorothalonil	2,4,5,6-Tetrachloroisophthalo- nitrile	See C-5
Chloroxuron	3-[p-(p-Chlorophenoxy)-phenyl]= 1,1-dimethylurea	See C-6
Chlorpropham	Isopropyl-N-(3-chloro-phenyl)= carbamate	See C-7
CIPC	See Chlorpropham	
Citcop	Unknown	
Citcop 4EC	See Citcop	
Cyanazine	2-[(4-Chloro-6-(ethylamino)-s= triazin-2-yl)amino]-2-methyl- propionitrile	See C-8
Cygon	See Dimethoate	
Cythion	See Malathion	
2,4-D	2,4-Dichlorophenoxyacetic acid	See D-1
Daconil 2787	See Chlorothalonil	
Dacthal	See DCPA	
2,4-D Amine	2,4-Dichlorophenoxyacetic acid, dimethylamine salt	See D-2
2,4-DB	4-(2,4-Dichlorophenoxy)= butyric acid	See D-3
DCPA	Dimethyl-2,3,5,6-tetra- chloroterephthalate	See D-4
Dibutalin	See Butralin	
Dicamba	2-Methoxy-3,6-dichlorobenzoic acid	See D-5

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Pesticide	Chemical Name	Structure*
Dichlobenil	2,6-Dichlobenzonitrile	See D-6
Dicofol	1,1-bis(p-Chlorophenyl)= 2,2,2-trichloroethanol	See D-7
Dimethoate	0,0-Dimethyl-S-(N-methylcarbamoylmethyl)-phosphorodithioate	See D-8
Diphenamid	N,N-Dimethyl-2,2-diphenylacetamide	See D-9
Dithane M-22	See Maneb	
Dithane M-45	See Mancozeb	
Dithane Z-78	See Zineb	
DO 14	See Propargite	
Dormart Oil	Unknown	
Dual	See Metolachlor	
Dyanap	See Naptalam	
Dymid	See Diphenamid	
Endosulfan	(Mixed Isomers) 6,7,8,9,10,10-Hexachloro-1,5,- 5a,6,9,9a-hexahydro-6,9-methano- 2,4,3-benzodioxathiepin-3-oxide	See E-1
Enide	See Diphenamid	
Eptam	See EPTC	
EPTC	S-Ethylidipropylthiocarbamate	See E-2
Eradicane	See EPTC	
Ethylparathion	See Parathion-ethyl	
Folpet	N-(Trichloromethylthio)-phthalimide	See F-1
Furloe	See Chlorpropham	
Gesatop	See Simazine	
Giberellic Acid	Unknown	
Glyphosate	N-(Phosphonomethyl)-glycine	See G-1
Gramoxone	See Paraquat-dichloride	
Guthion	See Azinphos-methyl	

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Pesticide	Chemical Name	Structure*
Heptachlor	1,4,5,6,7,8,8-Heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene	See H-1
Hexachlorobenzene	Hexachlorobenzene	See H-2
Imidan	See Phosmet	
Kelthane	See Dicofol	
Lannate	See Methomyl	
Lasso	See Alachlor	
Lexone	See Metribuzin	
Malathion	Diethylmercaptosuccinate, s-ester with O,O-dimethylphosphorodithioate	See M-1
Mancozeb	Unknown	
Maneb	Manganous ethylene-1,2-bis-(dithiocarbamate) [polymeric]	See M-2
Manzate	See Maneb	
Marlate	See Methoxychlor	
MCPA	(4-chloro-2-methylphenoxy)=acetic acid	See M-3
MCPP	2-(4-Chloro-2-methylphenoxy)=propionic acid	See M-4
Mediben	See Dicamba	
Metasystox R	See Oxydemeton-methyl	
Methomyl	S-Methyl-N-[(methylcarbamoyl)-oxy]-thioacetimidate	See M-5
Methoxychlor	di-(p-Methoxyphenyl)-trichloromethylmethane, mixed p,p' and o,p' isomers	See M-6
Metriram	Zinc-activated polyethylene-thiuramdisulphide	See M-7
Metolachlor	2-Chloro-N-(2'-ethyl-6'-methylphenyl)-N-(2-methoxy-1-methylethyl)-acetamide	See M-8
Metribuzin	4-Amino-6-[1,1-dimethyl(ethyl)]=3-(methylthio)-1,2,4,-triazin-5=(4H)-one	See M-9

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Pesticide	Chemical Name	Structure*
NAA	1-Naphthalene acetic acid	See N-1
Naphthalene acetic acid	See NAA	
Naptalam	Sodium salt of-N-1-Naphthyl-phthalimide	See N-2
N-serve	Unknown	
NU-Film-17	See Pinolene	
Octachlor	See Chlordane	
Omite	See Propargite	
Orthocide 406	See Captan	
Oxydemeton-methyl	S-[2-(ethylsulfinyl)-ethyl]= 0,0-dimethylphosphorothioate	See O-1
Paraquat-dichloride	1,1'-Dimethyl-4,4'-bipyridilium- dichloride	See P-1
Parathion	See Parathion-ethyl	
Parathion-ethyl	0,0-Diethyl-o-p-nitrophenyl- phosphorothioate	See P-2
Penoxalin	N-(1-Ethylpropyl)-3,4-dimethyl= 2,6-dinitrobenzenamine	See P-3
Phaltan	See Folpet	
Phosmet	0,0-Dimethyl-s-phthalimido- methylphosphorodithioate	See P-4
Pinolene	Unknown	
Plyac	Unknown	
Polynam	See Metiram	
Prefar	See Bensulide	
Princep	See Simazine	
Profluralin	N-(Cyclopropylmethyl)- α,α,α = trifluoro-2,6-dinitro-N= propyl-p-toluidine	See P-5
Propachlor	2'-Chloro-N-isopropylacetanilide	See P-6
Propargite	2-(p-tert-Butylphenoxy)-cyclo- hexyl-2-propynyl sulfite	See P-7
Prowl	See Penoxalin	

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Pesticide	Chemical Name	Structure*
Pyrethrum	Unknown	
Ramrod	See Propachlor	
Ravage	See Buthidazole	
Roundup	See Glyphosate	
Security	See Parathion-ethyl	
Sencor	See Metribuzin	
Sevin	See Carbaryl	
Simazine	2-Chloro-4,6-bis(ethylamino)= 1,3,5-triazine	See S-1
Sutan	See Butylate	
2,4,5-T	2,4,5-trichlorophenoxyacetic acid	See T-1
Tenoran	See Chloroxuron	
Tetrachloroterephthalic acid	Tetrachloroterephthalic acid	See T-2
Thiodan	See Endosulfan	
Tolban	See Profluralin	
Toxakil	See Toxaphene	
Toxaphene	A mixture of chlorinated cam- phenes of uncertain identity	
Treflan	See Trifluralin	
Trifluralin	α,α,α -Trifluoro-2,6-dinitro= N,N-dipropyl-p-toluidine	See T-3
Velsicol 58-CS-11	See Dicamba	
Velsicol 1068	See Chlordane	
Weedone	See 2,4-D	
Zineb	Zinc ethylene-1,2-bis-(dithio- carbamate) [polymeric]	See Z-1

* Refer to structures on following pages.

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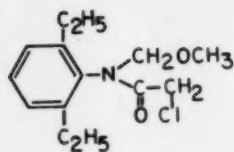
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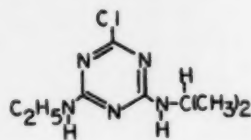
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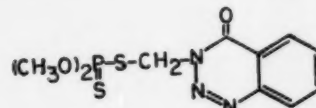
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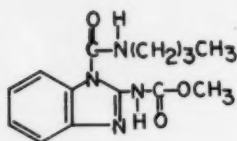
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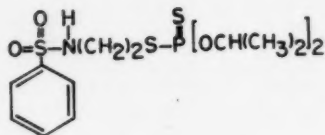
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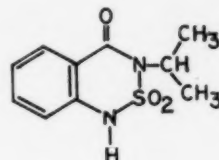
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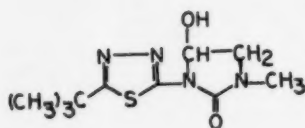
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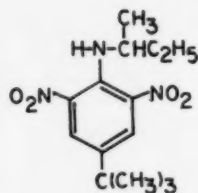
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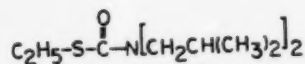
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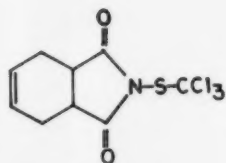
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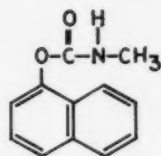
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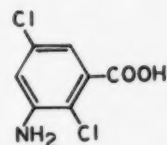
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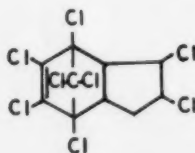
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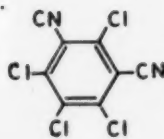
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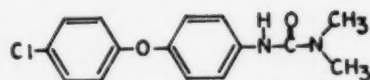
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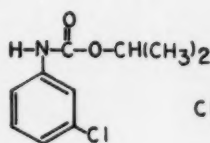
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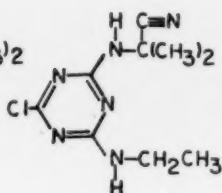
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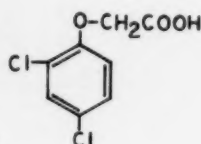
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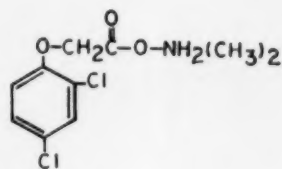
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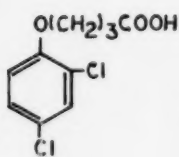
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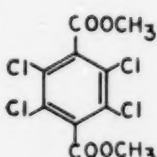
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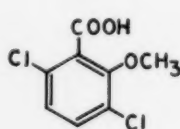
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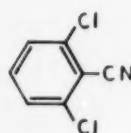
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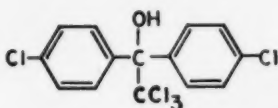
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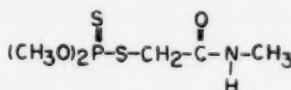
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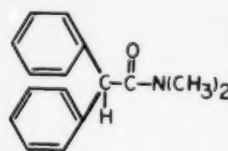
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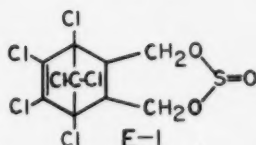
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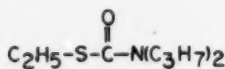
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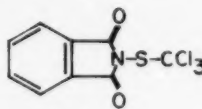
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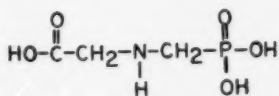
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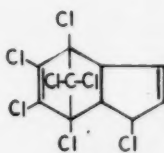
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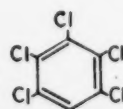
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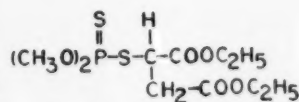
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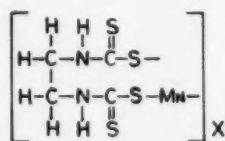
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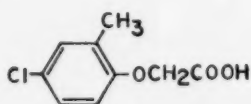
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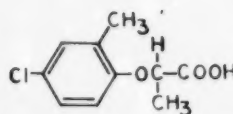
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Horticulture Macropit--

Core samples of soil of about 100 g size to a depth of 10 cm and liquid samples of 500 ml were taken initially from eight sampling points spaced uniformly across the pit surface. Each sample was then solvent extracted individually and the pesticides and metabolites were separated and quantitated. The data from these individual samples established unequivocally the extreme segregation of the pesticides deposited in this pit. Variations in concentration at the eight sampling points was dramatic as shown by the results tabulated in Table 2. This conclusion is further verified by the data in Table 3 where the range in concentration observed for several analyses in 1977 prior to October is reported. The wide range in concentration is due to uneven deposition of the waste pesticides and their apparent immobility in both the soil and the liquid phase.

To establish whether a composited sample would give results equivalent to the average of the individual analyses, eight samples were taken in October 1977. A portion of each sample was formed into a single composite and all samples were then analyzed. The results are tabulated in Table 4 where the analysis of the composited sample agrees very well with the average of the eight individual analyses. Therefore all samples after October 1977 were composited. The individual analyses were abandoned.

Water samples were also collected from the drainage tile surrounding this concrete lined pit. Other water samples were taken from sites remote from the pit location.

Air samples for pesticide vapor analyses were collected in the vicinity of the pit using a vacuum pump for drawing the desired air volumes through a sorption column containing ten grams of the macroreticular resin, XAD-2

Horticulture Micropits--

Soil and liquid samples were removed periodically from the four micropits located at the Horticulture farm. The rocks were completely removed prior to taking the sediment sample by coring. The liquid samples were taken in much the same manner as that described later on for the minipits.

Agronomy Macropits--

Water samples only were collected from five access tiles at the Agronomy macropit #2. These were analyzed individually and the pesticide concentrations varied widely as shown by the data tabulated in Table 5. Equal portions of these same samples were composited and also analyzed. The results for the composited sample and the average of the individually analyzed samples are listed in Table 6 for comparison. As with all the other pit systems under investigation, these results in Tables 5 and 6 establish the heterogeneous nature of the pesticide concentrations within any one pit system and the validity of composite sampling.

No soil samples were taken from within the Agronomy pit. One set of soil samples to a depth of 3 meters was taken from 10 cm diameter holes augered at each outside corner of macropit #2. Water samples from outside the pit were

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TABLE 2. DATA SHOWING THE RANGE IN PESTICIDE CONCENTRATION AT THE
EIGHT SAMPLING POINTS IN THE HORTICULTURE DISPOSAL PIT - OCTOBER 1977

Pit at Various Sampling Points

Pesticide	Sample Type	#1	#2	#3	#4	#5	#6	#7	#8
Azinphos-Methyl	H ₂ O Soil	0.3 384	5.5 94	0.9 157	4.4 2902	1.1 765	4.5 84	0.6 396	0.2 24
Chlorothalonil	H ₂ O Soil	0.2 108	1.2 10328	0.2 25	1.1 287	<0.1 298	0.7 29	1.0 77	1.5 177
DCPA	H ₂ O Soil	0.9 492	1.0 639	0.5 8.5	1.7 9738	0.8 984	0.7 72	1.2 1026	0.5 38
Dichlobenil	H ₂ O Soil	<0.1 1.2	<0.1 1.1	<0.1 0.9	<0.1 2.4	0.1 1.9	<0.1 0.4	0.1 4.2	0.1 0.3
Endosulfan I	H ₂ O Soil	0.4 42	0.3 13	0.2 4.2	0.8 60	0.4 21	0.4 1.2	0.3 3.8	0.2 1.9
Endosulfan II	H ₂ O Soil	0.2 18	0.2 9.8	<0.1 2.0	0.5 37	0.3 9.8	0.2 1.9	0.2 3.4	<0.1 <0.1
EPTC	H ₂ O Soil	12 8.1	7.6 6.2	1.2 3.3	19 11	10 5.1	7.2 3.2	19 1.7	9.4 <0.1
Hexachlorobenzene	H ₂ O Soil	<0.1 50	<0.1 85	<0.1 0.7	0.2 77	<0.1 74	<0.1 8.6	0.2 124	<0.1 6.5
Phosmet	H ₂ O Soil	<0.1 <0.1	2.0 <0.1	<0.1 <0.1	5.4 1.6	1.6 8.6	2.8 1.7	<0.1 <0.1	<0.1 <0.1
Propachlor	H ₂ O Soil	9.2 <0.1	7.1 <0.1	4.4 <0.1	36 <0.1	15 <0.1	1.8 <0.1	28 <0.1	4.3 <0.1

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TABLE 3. RANGE OF PESTICIDE CONCENTRATIONS FROM EIGHT SAMPLING POINTS
IN HORTICULTURE MACRO DISPOSAL PIT - 1977

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Pesticide	In	PPM Range			
		March	June	August	October
Azinphos-Methyl (Guthion)	H ₂ O	1.5 - 8.1		0.2 - 0.8	0.2 - 4.5
	Soil	13 - 3767	20 - 916	16 - 1508	24 - 2902
Chlorothalonil (Bravo)	H ₂ O	0.0 - 0.5	0.1 - 1.4	0.1 - 0.2	0.2 - 1.5
	Soil	19 - 4585	30 - 593	14 - 902	25 - 10328
DCPA (Dacthal)	H ₂ O	0.2 - 0.4	0.1 - 6.3	0.3 - 1.0	0.5 - 1.7
	Soil	32 - 3548	4 - 13376	13 - 4679	38 - 1026
Propachlor (Ramrod)	H ₂ O	8 - 58	21 - 159	56 - 105	4 - 28
	Soil		9 - 163	14 - 22	
Hexachlorobenzene	H ₂ O	0.1 - 0.3	0.1 - 50	0.1 - 0.2	0.0 - 0.2
	Soil	1 - 202	4 - 300	1 - 307	7 - 124
Endosulfan I (Thiodan)	H ₂ O		0.0 - 0.6	0.0 - 0.3	0.2 - 0.8
	Soil	2 - 104	1 - 27	3 - 45	2 - 42
Endosulfan II (Thiodan)	H ₂ O		0.0 - 0.3	0.0 - 0.1	0.1 - 0.5
	Soil	2 - 67	1 - 11	7 - 24	2 - 37
Phosmet (Imidan)	H ₂ O			0.1 - 2.5	1.6 - 5.4
	Soil			8 - 51	
Dichlobenil (Casoron)	H ₂ O				0.0 - 0.1
	Soil				
Captan	H ₂ O			0.0 - 0.1	8 - 21
	Soil				11 - 15

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TABLE 4. AMOUNTS OF PESTICIDES IN PPM
IN HORTICULTURE MACRO DISPOSAL PIT - OCTOBER 1977

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Pesticide	Sample Type	Ave. of 8 Samples	Composite Sample
Azinphos-methyl	H ₂ O	2.2	2.2
	Soil	603	601
Chlorothalonil	H ₂ O	0.9	0.8
	Soil	1438	1416
DCPA	H ₂ O	0.9	0.9
	Soil	534	525
Dichlobenil	H ₂ O	0.1	0.1
	Soil	1.6	1.5
Endosulfan I	H ₂ O	0.4	0.4
	Soil	19	19
Endosulfan II	H ₂ O	0.2	0.2
	Soil	11	11
EPTC	H ₂ O	12	11
	Soil	5.0	4.8
Hexachlorobenzene	H ₂ O	0.1	0.1
	Soil	54	55
Phosmet	H ₂ O	1.8	1.1
	Soil	1.7	1.1
Propachlor	H ₂ O	14	13
	Soil	<0.1	<0.1

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TABLE 5. DATA SHOWING THE RANGE IN HERBICIDE CONCENTRATION AT THE FIVE SAMPLING POINTS IN THE AGRONOMY DISPOSAL PIT - JUNE 1978

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Pesticide	PPM at Various Sampling Points				
	#1	#2	#3	#4	#5
Alachlor	1646	4681	819	1347	1667
Atrazine	54	146	22	50	10
Butylate	200	130	24	81	125
Chlorpropham	110	210	25	193	314
Cyanazine	220	843	110	235	284
Dimethoate	<1	4	3	<1	4
EPTC	44	38	24	40	50
Propachlor	226	442	144	306	369
Trifluralin	1395	370	989	2372	3256

TABLE 6. AMOUNTS OF HERBICIDES IN PPM AT FIVE SAMPLING POINTS IN THE AGRONOMY DISPOSAL PIT - JUNE 1978

Pesticide	Ave. of 5 Samples	Composite Sample
Alachlor	2032	2014
Atrazine	68	.60
Butylate	112	111
Chlorpropham	170	102
Cyanazine	338	258
Dimethoate	2	3
EPTC	39	41
Propachlor	297	288
Trifluralin	1676	1791

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also collected from the seepage of ground water into the augered holes and from other sites remote from the pit location.

Air samples for pesticide vapor analyses were collected in the vicinity of the Agronomy pits using a vacuum pump and XAD-2® as described earlier.

No samples were taken from the Agronomy macropit #1 which had been abandoned prior to the inauguration of this research program. Some soil core and water samples were taken in the immediate vicinity of this abandoned pit and compared to ambient samples taken at sites remote from the disposal pits.

Agronomy Minipits--

A controlled field experiment was designed to determine if selected pesticides would degrade in small inexpensive disposal pits. Atrazine (Aatrex®; 80W), alachlor (Lasso®; E.C.), 2,4-D ester (Weedone LV4®; E.C.) and trifluralin (Treflan®; E.C.) were spiked into 110 liter plastic garbage containers buried partially in the ground and filled with 60 liters of water and 15 kilograms of soil. The degradation of these herbicides were studied individually and as a mixture at the two concentrations of 0.5 and 0.025% active ingredient. Additional variables of aeration at one liter per minute and peptone nutrients at 0.1% by weight resulted in a factorial experiment of 40 systems. Representative 100 gram samples of the soil and liquid contents of all minipits were taken for analyses at intervals of 0, 1, 3, 4, 8, 12, 16, 20, 24, 28, 52, 68 and 123 weeks after addition. The aliquots were obtained by slowly lowering and raising a four ounce bottle, capped with a two hole rubber stopper, through the vortex formed by vigorous mixing of the contents of the container with a propeller blade attached to a shaft driven by a variable speed drill. The amount of water necessary to adjust the volume to zero weeks was recorded before each sample collection. The pH measurements were made immediately after each sample collection. The temperatures of the pits were also measured and recorded on an intermittent basis.

Sample collection and analyses similar to that described above were also accomplished by personnel from Entomology for the two insecticides, carbaryl and parathion. For analytical details for these two insecticides, see the section on entomological studies.

Extraction Procedures - Macropits

The procedures employed for the liquid, soil, water and air samples are described and discussed below.

Liquid Samples--

Three procedures were evaluated for extracting pesticides from liquid samples collected at the Horticulture and Agronomy macropits. They were the resin sorption method of Junk et al. (1974), solvent extraction with hexane-diethylether, and solvent extraction with methylene chloride. Comparable recoveries were achieved for all three procedures. The methylene chloride procedure was chosen because of the ready availability of pure solvent and

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the simplicity of the method.

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Liquid samples collected at the Agronomy macropit #2 were stabilized suspensions which required pretreatment prior to the extraction. The pretreatments which were investigated were 24 hr settle, 24 hr settle plus one hr centrifuging at 2500 rpm, and 24 hr settle plus one hr centrifuging plus filtering through a 1 to 2 micron filter. These three methods were used for the liquid samples collected on August 29 and September 27 (1978). The results are tabulated in Table 7. For most of the pesticides comparable results were obtained with the amounts after centrifuging and filtering generally being lower. These samples also had the lowest turbidity and thus were more representative of true dissolved constituents. All samples collected after September 27, 1978 were settled for 24 hr and then centrifuged and filtered before extraction.

Soil Samples--

Five methods of extraction were evaluated for soil samples collected at the Horticulture macropits. The results for this evaluation are tabulated in Table 8. The Woolson (1974) procedure was selected for use throughout the course of our investigations of all soil and sediment samples.

Water Samples--

The ground water samples from sites adjacent to and remote from the disposal pit locations and the well water samples were collected in gallon sized amber bottles which were then capped with a Teflon® liner and transported to the laboratory for extraction and analyses of pesticide residues. The procedure employed was the resin sorption method using XAD-2 as described by Richard et al. (1975) and Junk et al. (1976) for measuring very low amounts of selected pesticides.

Air Samples--

The atmospheres in the vicinity of the pits were sampled for pesticide vapors using a macroreticular resin, XAD-2, and a vacuum pump. Collection efficiencies for some of the pesticides determined by this method were measured in the laboratory using simulated atmospheres. These test results, given in Table 9, show the probable validity of this method for pesticide vapors in the atmosphere above the pits and upwind from the disposal sites.

Extraction Procedures - Minipits

The combined sediment and water samples collected from the minipits were centrifuged for 1/2 hr at $\approx$ 2000 rpm. The water was decanted and the volume measured prior to transfer to a 250 ml separatory funnel followed by extraction four times with 50 ml each of diethyl ether for the high concentration samples and with 50 ml followed by two 25 ml volumes for the low concentration samples. The soil portion was extracted by adding 50 ml of diethyl ether to the residue in the sample bottle which was then capped and agitated for 15 minutes on a wrist action shaker. The diethyl ether was decanted and the high concentration samples were extracted three more times with 50 ml of diethyl ether by hand shaking the capped bottles for 2 to 3

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TABLE 7 . VARIATION IN HERBICIDE CONCENTRATION WITH
SETTLING, CENTRIFUGING AND FILTRATION

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Herbicide	PPM on Aug 29			PPM on Sep 27		
	24h Settle	Centrifuge	Centrifuge + Filter	24h Settle	Centrifuge	Centrifuge + Filter
EPTC (Eptam)	7	4	2	7	6	5
Butylate (Sutan)	13	7	3	9	8	4
Chlorpropham (Furloe)	15	8	6	12	10	8
Propachlor (Ramrod)	132	110	32	152	144	140
Atrazine (Aatrex)	279	193	174	14	12	11
Trifluralin (Treflan)	189	108	51	117	101	42
Dimethoate (Cygon)	1.1	0.9	0.4	*	-	-
Alachlor (Lasso)	318	223	171	374	340	242
Cyanazine (Bladex)	97	119	109	166	138	148
Metribuzin (Sencor)	111	100	138	-	-	-

* Not measured

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TABLE 8. COMPARISON OF VARIOUS SOLVENTS AND EXTRACTION PROCEDURES
FOR RECOVERY OF PESTICIDES FROM A HORTICULTURE PIT SAMPLE

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Pesticide	PPM by Method [†]				
	A	B	C	D	E
Bensulide	26	16	22	24	24
Chlorothalonil	162	184	193	196	146
DCPA	1116	1141	1129	1054	1017
Dichlobenil	4	2	3	5	4
Endosulfan I	38	37	36	36	35
Endosulfan II	23	23	22	22	21
Hexachlorobenzene	89	91	89	82	79
Methoxychlor	93	95	93	93	91
Phosmet	4	2	3	3	2
Trifluralin	36	30	22	20	22
2,4-D acid	*	*	*	*	*
2,4,5-T acid	*	*	*	*	*

[†]A = batch extraction using hexane-acetone according to procedure by Woolson (1974)

B = ultrasonic extraction using isopropyl alcohol

C = ultrasonic using hexane-acetone

D = ultrasonic using benzene-isopropyl alcohol

E = soxhlet extraction using hexane-acetone

*Not detected

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TABLE 9. COLLECTION EFFICIENCIES OF PESTICIDE VAPORS
USING XAD-2 FOR AIR SAMPLING*

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Pesticide	Vol. in liters	Flow in l/min	Collection† Efficiency, %
chlorothalonil	200	3	100
trifluralin	200	3	100
trifluralin	700	10	77‡
dacthal	200	3	100
dacthal	700	10	95
alachlor	200	3	99
butylate	200	3	97
EPTC	200	3	96
EPTC	700	10	107
endosulfan	200	3	115
malathion	200	3	52
hexachlorobenzene	200	3	66
hexachlorobenzene	3000	50	58
2,4-D(isopropyl ester)	200	3	98
dichlobenil	200	3	99
aroclor 1254	200	3	81
aroclor 1254	700	10	88
methoxychlor	700	10	98

*Total amount of each pesticide was 100 micrograms.

†Average of two runs.

‡87% recovery achieved for 10 micrograms of treflan.

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minutes. The low concentration samples were extracted two additional times with 25 ml of diethyl ether. The pesticides in these extracts were determined by gas chromatography. The soil residues were weighed after drying overnight at 105°C.

The 2,4-D samples were acidified with 0.2N H₂SO₄ before extracting. Aliquots of the 2,4-D extracts were esterified with diazomethane to determine the amounts of 2,4-D acid.

This extraction procedure for the minipit samples was checked for efficiency of the recovery of model pesticides from combined 10 g soil and 100 ml water samples. Atrazine, alachlor, trifluralin and 2,4-D ester were spiked into water and soil as Aatrex®, Lasso®, Treflan® and Weedone® respectively. At the levels of 0.025 and 0.5% active ingredient the average recoveries were 97 and 96% respectively. Comparable efficiencies were obtained for mixtures of these four herbicides.

Separation and Detection Procedures

The majority of the pesticides were separated by gas chromatography and detected by a variety of general and specific detectors such as flame ionization, electron capture, flame photometric, electron impact and chemical ionization mass spectrometric and nitrogen-phosphorus. Two columns were most useful for separating the pesticides in the extracts of the samples collected from the Horticulture and Agronomy disposal pits. These two columns were a 4% SE-30/6% OV210 and a 10% DC-200. A 30 meter SE-54 glass capillary column was employed for more complete separations when necessary to aid in identification and analyses procedures. Relative retention times of several pesticides and methyl esters are given in Table 10 for the SE-54 glass capillary column and in Table 11 for four packed columns.

For those compounds which could not be measured on the above columns using gas chromatography, the special handling procedures discussed below were devised and used.

Special Handling--

Some of the pesticides deposited in the disposal pits were not gas chromatographable directly. These required special treatment to give products that could be determined by gas chromatography or by other procedures. These pesticides and the procedures employed for their analyses are discussed below.

Benomyl--The extraction and isolation procedure of Austin and Briggs (1976) was used. After isolation and concentration benomyl and its metabolites were determined colorimetrically.

Dithiocarbamates--Includes mancozeb, maneb, polyram and dithane. The head space procedure for screening food samples for dithiocarbamate pesticide residues by McLeod and McCully (1969) was adapted to the soil and water samples.

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TABLE 10. RELATIVE RETENTION TIMES OF VARIOUS PESTICIDES
AND METHYL ESTERS (M.E.) ON A SE-54 CAPILLARY COLUMN*

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Pesticide	t _R
Trimethylphosphate	0.09
Dimethylthiophosphate	0.18
Dimethyldithiophosphate	0.22
2,4-Dichlorophenol-M.E. [†]	0.33
Linuron	0.34
Dichlobenil	0.42
Eptam	0.43
2,6-Dichlorobenzoic Acid-M.E.	0.45
Tetrahydrophthalimide-M.E.	0.49
Butylate	0.50
2,4,5-Trichlorophenol-M.E.	0.51
Tetrahydrophthalimide	0.59
Dicamba-M.E.	0.63
MCPA-M.E.	0.63
MCPP-M.E.	0.64
Propachlor	0.71
Chlorpropham	0.74
2,4-D-M.E.	0.74
BHC	0.74, 0.80
Trifluralin	0.78
Pentachlorophenol-M.E.	0.79
Hexachlorobenzene	0.79
Lindane (γ)	0.81
Dealkylalachlor	0.81
Chloramben-M.E.	0.82
Benzimidazole	0.83
Atrazine	0.84
Diamino treflan	0.85
Simazine	0.85
Profluralin	0.85

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TABLE 10. (continued)

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Pesticide

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Dimethoate

0.85

Nitroamino treflan

0.87

Dinitramine

0.88

Fluchloralin

0.88

2,4,5-T-M.E.

0.89

Chlorothalonil

0.92

Bentazon-M.E.

0.93

Metribuzin

0.95

Endosulfan I

0.96

Alachlor

0.97

Chloroxuron

0.98

Aldrin

1.00

Metolachlor

1.02

Malathion

1.03

DCPA

1.04

Ethyl Parathion

1.05

Heptachlor Epoxide

1.07

Cyanazine

1.08

Butralin

1.09

Penoxalin

1.09

Captan

1.11

Folpet

1.11

Kelthane

1.15, 1.21

p,p'-DDE

1.20

Dieldrin

1.22

Endosulfan II

1.22

p,p'-DDD

1.27

p,p'-DDT

1.33

Methoxychlor

1.42

Naptalam-M.E.

1.47

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*Temperature programmed at 5°/min from 80-270°C

†M.E. = methyl ester

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TABLE 11. RELATIVE RETENTION TIMES ON VARIOUS STATIONARY PHASES
OF SELECTED PESTICIDES DEPOSITED IN HORTICULTURE MACROPIT

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Pesticide	10% DC-200	4% SE-30/ 6% OV-210	5% OV-210	1.5% OV-17/ 1.95% OV-210
Dichlobenil	.14	.22	.38	0.21
Propachlor	.21	.50	.86	0.46
Atrazine	.39	.66	.81	0.66
Carbaryl	.44	-	.21	0.28
Alachlor	.72	1.04	-	1.02
Heptachlor	.75	.82	.86	0.83
Malathion	.76	1.30	3.00	1.39
Ethyl Parathion	.87	1.67	3.67	1.61
Keltane	.92	1.25	1.90	1.46
DCPA (Dacthal)	.93	1.46	2.11	1.39
Hydroxychlorthane	.94	1.37	1.37	1.31
Captan	1.10	1.83	3.33	2.36
Heptachlor Epoxide	1.17	1.40	1.86	1.50
Sulfur	1.23	-	.88	-
Endosulfan I	1.49	1.72	2.40	1.94
Dieldrin	1.73	2.01	2.79	2.24
P,P'DDE	1.73	1.75	1.89	2.08
Endosulfan II	1.95	2.57	4.10	3.35
P,P'DDD	2.07	2.25	3.29	3.14
O,P'DDT	2.19	2.39	2.38	2.86
P,P'DDT	2.66	3.12	3.48	3.68
Phosmet	3.25	-	-	8.89
Methoxychlor	3.70	3.98	5.18	
Guthion	3.87	7.60	15.80	
Chlordane	.70	.78	.86	
	.79	.84	1.76	
	.93	1.06		
	1.10	1.50		
	1.33	1.64		
	1.47			

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Paraquat--The extraction and gas chromatographic procedure of King (1978) was employed on water and soil samples.

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Acidic pesticides and metabolites--These were concentrated from aqueous solution by the anion procedure of Richard and Fritz (1980). The anionic materials in these concentrates were methylated using diazomethane and the derivatized products were separated and detected by gas chromatography. Test results of the recovery efficiencies by this method for several pesticides and suspected metabolites are given in Table 12.

RESULTS AND DISCUSSION

Horticulture Macropit

The accumulations of the amounts of pesticides deposited in the Horticulture macropit are given in Table 13 for the period June 6, 1977 to October 1, 1979. If more detailed accumulation data are needed for interpretation of the analytical results presented here, they may be found elsewhere in this report.

Even though a very conscientious effort was made to record all depositions, this pit is referred to as an uncontrolled system since the amounts and the timing of the dumps were at the discretion of the farm operator rather than the analytical chemist. For example, an appreciable amount of Polyram was deposited prior to June 6, 1977 and none was dumped from June through October 10. Sometime after October 1977 but before May 1978, an additional amount was dumped to bring the total to 2392 g. However the late growing seasons of 1978 and 1979 were different than 1977 because some further dumps of polyram were made during the summer and fall. Thus the analytical data for the various pesticides will be a perturbation of the total amount deposited, the timing of the dumps and the sampling, the chemical decay, the biological decay and losses due to incomplete containment. Under this kind of operational and analyses mode the best opportunity to document the feasibility of the disposal system is to review the analytical data for pesticides such as Dacthal, Ramrod, Captan, Imidan, Polyram, etc. which have been deposited in large amounts continuously over a three year period. The absence of any appreciable build-up is a first order indication of feasibility.

Soil and Liquid Analyses--

The amounts of the pesticide residues in the soil and water samples taken from the macropit at the Horticulture farm are tabulated in Table 14. The numerical data for all of 1977 represent the average of eight separate analyses of samples taken from eight positions within the pit. For 1978 and 1979 the samples from the eight positions were composited prior to the extraction and analyses. None of the many pesticides being deposited appear to be building up in concentration although no appreciable dumps of pesticides known to be highly recalcitrant have been dumped into the pit over the past three years.

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The behavior of the individual pesticides is best shown by the plots in

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TABLE 12. RECOVERY OF SELECTED PESTICIDES AND METABOLITES
SPIKED INTO WATER AT 200 PPB AND EXTRACTED BY ANION EXCHANGE PROCEDURE

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Pesticide or Metabolite	% Recovered
3-Isopropyl-1H-benzo-2,1,3-thiadiazin-4-one-2,2-dioxide (Basagran)	91
3-Amino-2,5-dichlorobenzoic acid (Chloramben)	86
2,4-Dichlorophenoxyacetic acid (2,4-D)	93
2-Methoxy-3,6-dichlorobenzoic acid (Dicamba)	98
N-(Phosphonomethyl)-glycine (Glyphosate)	103
2-(4-Chloro-2-methylphenoxy)-propionic acid (MCPP)	93
Sodium salt of N-1-naphthylphthalimide (Naptalam)	95
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	102
2,4-Dichlorophenol	100
2,4,5-Trichlorophenol	95
Pentachlorophenol	100
2,4-Dichlorobenzoic acid	91
Tetrachloroterephthalic acid	83
Dimethylphosphoric acid	76
Dimethylthiophosphoric acid	94
4-Cyclohexene-1,2-dicarboximide	95
Average % Recovery	= 93

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TABLE 13. DISPOSAL ACCUMULATIONS AT THE HORTICULTURE MACROPIT
IN GRAMS OF ACTIVE INGREDIENT (AI)

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Pesticide Deposited	Grams AI at Listed Dates					
	Jun 6 1977	Oct 10 1977	May 31 1978	Nov 3 1978	Jun 1 1979	Oct 1 1979
Polyram*	1974	1974	2392	2699	3108	3244
Imidan*	730	1651	1718	2319	2535	2716
Captan*	1050	2050	2126	2616	2678	3338
Citcop*	58	635	635	1895	2033	2557
Thiodan*	42	753	753	1009	1009	1027
Paraquat*	29	132	184	249	470	522
Ramrod*	1335	2273	2567	4495	4495	5435
Bravo*	6	619	619	1752	1752	2275
Marlate*	39	108	147	2242	2328	2427
Tenoran*	569	815	815	1381	1381	1381
Sevin*	2	402	402	991	991	1200
NAA	0	<1	<1	<1	<1	<1
N-Serve	0	<1	<1	<1	<1	<1
Dymid	229	1367	1367	3113	3113	3113
Treflan	138	138	160	160	569	569
Benlate*	12	14	35	271	302	337
Guthion*	12	14	14	30	30	30
Prowl	4	4	4	4	4	4
Lasso	4	4	4	4	4	4
Amex	13	13	13	13	13	13
Dacthal*	3379	7926	8011	10340	10381	12167
Simazine	129	129	160	160	414	414

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TABLE 13. (continued)

Pesticide Deposited	Grams AI at Listed Dates					
	Jun 6 1977	Oct 10 1977	May 31 1978	Nov 3 1978	Jun 1 1979	Oct 1 1979
Eptam	975	975	975	1127	1127	1127
Dipel		<1	<1	<1	<1	<1
Lannate*		57	57	221	221	221
Dithane M-45		274	274	735	735	735
Malathion*		245	245	591	591	633
Kelthane*		386	386	386	386	387
Omite*		32	32	32	32	57
Roundup			44	44	44	44
Casoron*			12	12	295	295
Alanap			2325	2325	2325	2325
Prefar			1237	1237	1237	1237
Folpet			136	240	240	240
Butoxone				22	22	35
Dithane M-22				154	154	154
2,4-D*				369	369	369
MCP				185	185	185
Dicamba				37	37	37
Aatrex 80W					37	37
Metasystox R						2
Sencor						27
TOTAL	= 10729	22993	27852	43463	45647	50920

*Unknown amounts of these pesticides and Amiben, Carbaryl, Chlordane, Cythion, Heptachlor, Maneb, Pyrethrum, Toxaphene and Zineb were deposited prior to the commencement of accurate record keeping beginning June 1977.

WPA-267 (Gm.)
(8-78)

TABLE 14 PESTICIDES IN SOIL AND WATER AT HORTICULTURE MACRO DISPOSAL PIT

Pesticide	In	Amount in PPM															
		Mar 5 1977	Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	Apr 17 1979	May 29 1979	Jun 28 1979	Sep 21 1979	
Alachlor (Lano)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Azinphos-Methyl (Orthion)	H ₂ O Soil	3.4 1157	-	0.5 335	2.2 600	0.7 1.6	0.7 -	-	-	-	-	-	-	-	-	-	
Beromyl	H ₂ O Soil	-	-	-	-	0.7	-	-	5.9	-	2.4	2.1	2.1	4.4	3.4	4.2	
Bensulide (Prefar)	H ₂ O Soil	-	-	-	-	-	3.1	-	3.2	2.8	3.5	4.3	5.8	6.2	3.8	5.5	
Butralin (Amex)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	5.0	-	-	1.1	-	
Caplan (Orthocide)	H ₂ O Soil	-	-	<0.1 2.5	<0.1 6	0.4 4.8	0.2 6	-	-	-	-	-	-	-	-	-	
Carbaryl (Sevin)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Chlorothalonil (Bravo)	H ₂ O Soil	0.3 1238	0.4 197	0.1 397	0.8 1416	8.5 975	0.2 32	0.2 63	0.6 90	1.6 154	0.2 101	0.3 131	0.2 123	0.2 76	0.1 26	0.4 31	
Chloroxuron (Tenoran)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	1.0 5.5	2.0 47	3.2 34	1.4 37	4.5 -	
2,4-D**	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	18	18	72	
2,4-DB	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
DCA (Dacthal)	H ₂ O Soil	0.3 1138	1.7 1780	0.5 982	0.9 529	19 823	0.9 291	1.9 972	1.8 903	6.3 670	1.1 1215	0.7 1278	0.5 1292	0.4 732	0.2 1285	0.7 1484	
Dicamba** (Banvel)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	5.0	2.1	-	
Dichlobenil (Casoron)	H ₂ O Soil	-	-	-	<0.1 1.5	<0.1 4.6	2.5 4.9	2.2 3.5	1.5 3.6	1.5 6.7	0.9 26	1.1 12	0.9 12	4.4 166	2.9 69	8.0 52	

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TABLE 14 (continued)

Pesticide	In	Amount in PPH														
		Mar 5 1977	Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978	May 1 1978	May 31 1978	Jun 29 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	Apr 17 1979	May 29 1979	Jun 28 1979	Sep 21 1979
Diphenamid (Dysid)	H ₂ O Soil	-	-	-	-	-	91 16	75 13	156 20	92 35	130 75	132 143	68 39	99 48	100 76	74 80
Endosulfan I (Thiodan)	H ₂ O Soil	<0.1 32	0.2 7.4	0.1 12	0.4 16	<0.1 16	0.3 12	0.6 14	0.4 14	0.9 20	0.4 24	0.4 30	0.1 37	0.1 16	0.1 30	<0.1 40
Endosulfan II (Thiodan)	H ₂ O Soil	<0.1 18	<0.1 3.9	<0.1 8.1	0.2 11	0.5 9.2	0.1 8.1	0.4 9.0	0.3 10	0.6 13	0.2 17	0.2 21	0.1 25	0.1 12	0.1 20	0.1 30
EPTC (EPTam)	H ₂ O Soil	-	-	-	10 4.7	6	1.0 1.9	0.4 1.3	2.6 2.8	1.0 2.4	1.8 3.8	0.8 5.6	1.5 4.0	<1 1	<1 1	* *
Ethylparathion	H ₂ O Soil	-	-	-	-	0.2 2.8	-	0.2 *	6	0.1 3.2	* 6.7	0.1 8.2	0.1 8	* *	* *	* *
Folpet	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Glyphosate (Roundup)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Hexachloro- benzene	H ₂ O Soil	0.2 61	6.4 50	<0.1 72	0.1 54	1.6 73	<0.1 23	0.1 37	<0.1 31	0.1 39	<0.1 66	<0.1 70	0.2 77	0.1 54	0.1 60	64
Kelthane	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Malathion (Cythion)	H ₂ O Soil	-	-	-	0.3 *	6 4.2	1.0 *	0.2 *	0.7 *	0.3 *	0.5 *	0.7 *	0.3 *	0.2 *	0.2 *	0.3
MCPP**	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Methoxy1	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Methoxychlor (Marlate)	H ₂ O Soil	-	-	-	-	1.6 44	0.2 36	0.6 46	0.7 138	2.8 135	0.7 108	0.7 154	0.7 122	0.1 79	0.1 115	0.2 191
Naphthalenecetic Acid	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

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Pesticide	In	Amount in PPM														
		Mar 5 1977	Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978	May 1 1978	May 31 1978	Jun 29 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	Apr 17 1979	May 29 1979	Jun 28 1979	Sep 21 1979
Naptalam** (Alanap)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	39	81	100	28
H ₂ O	Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N-Serve	Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Omite	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Paraquat 'dichloride	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Penoxalin (Prowl)	H ₂ O Soil	-	-	-	*	*	*	*	*	*	*	*	*	*	*	*
Phosmet (Imidan)	H ₂ O Soil	-	-	0.5	1.1	1.4	0.1	2.4	*	1.1	0.3	1.3	0.6	0.4	0.1	0.2
Polyram***	H ₂ O Soil	-	-	12	1.1	4.7	*	*	*	*	*	*	13	2.3	4.8	3.9
Propachlor (Ramrod)	H ₂ O Soil	-	-	-	1.9	1.4	-	2.6	3.7	3.1	6.3	6.7	*	2.1	1.9	*
Sinazine + Atrazine	H ₂ O Soil	-	-	-	49	120	29	72	134	149	307	363	270	167	404	237
Tetrachlorotere- phthalic Acid**	H ₂ O Soil	21	91	74	12	11	5.5	18	88	41	15	11	2.7	74	89	31
2,4,5-T**	H ₂ O Soil	-	28	13	13	6.0	*	1.1	13	7.3	*	3.5	5.1	45	30	24
Trifluralin (Treflan)	H ₂ O Soil	-	-	-	-	5.2	23	11	*	29	11	15	*	*	*	16
Metribuzin (Sencor)	H ₂ O Soil	-	-	-	-	*	*	*	*	2.1	*	*	*	*	*	15
		-	-	-	-	-	-	-	-	-	-	-	5.7	18	31	18
		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		-	-	-	-	-	-	-	-	-	-	-	-	-	0.1	0.2
		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
		-	-	-	-	0.4	0.3	0.8	*	*	*	*	*	1.1	*	0.8
		-	-	-	-	11	*	*	2.0	2.4	*	*	39	23	29	30
		-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.5
		-	-	-	-	-	-	-	-	-	-	-	-	-	-	25
**Manganese and Naphcozeb included in Polyram assay - Not measured																

**Not detected

**Measured as methyl ester

***Maneb and Mancozeb included in Polyram assay

- Not measured

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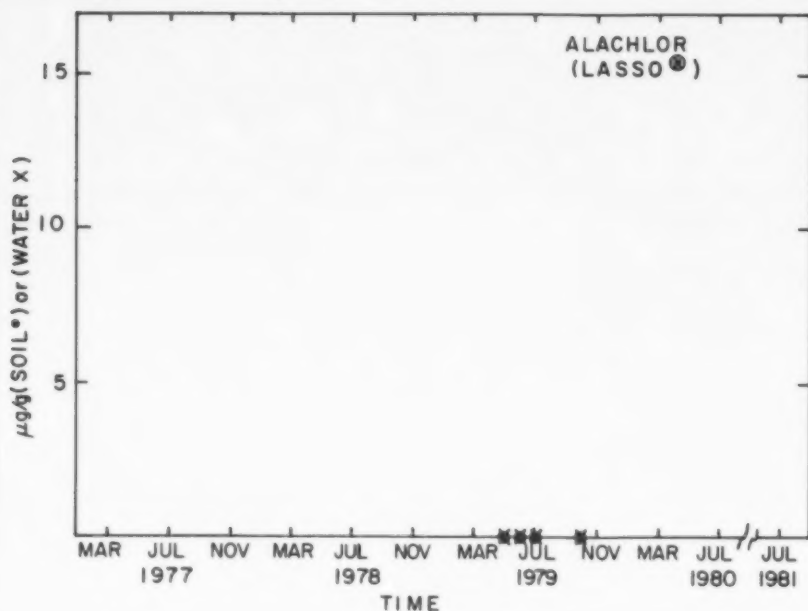
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Figure 13. Alachlor concentration in soil and water samples from horticulture macropit.

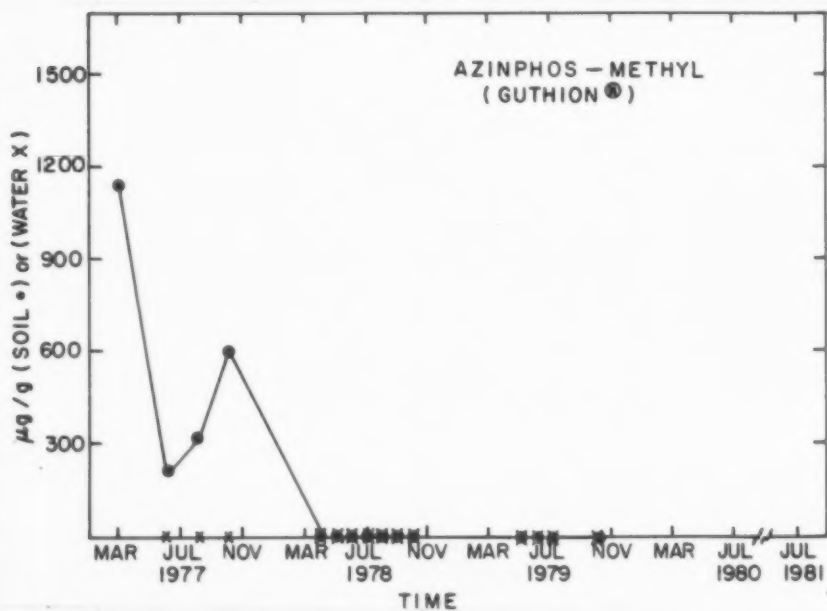


Figure 14. Azinphos concentration in soil and water samples from horticulture macropit.

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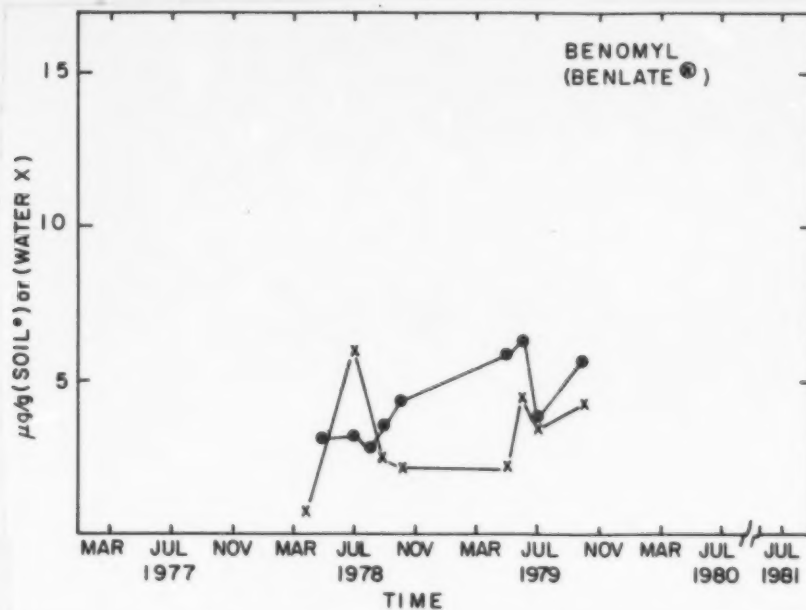


Figure 15. Benomyl concentration in soil and water samples from horticulture macropit.

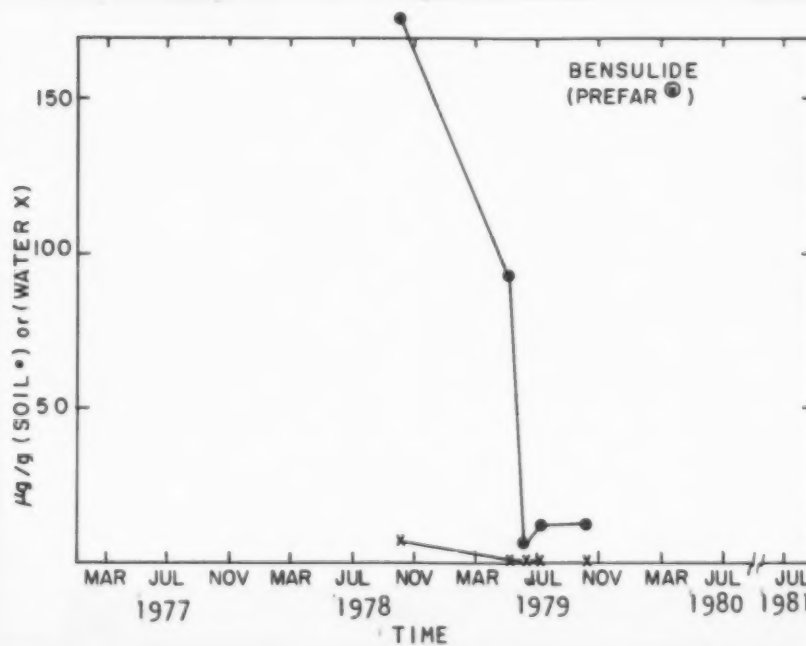


Figure 16. Bensulide concentration in soil and water samples from horticulture macropit.

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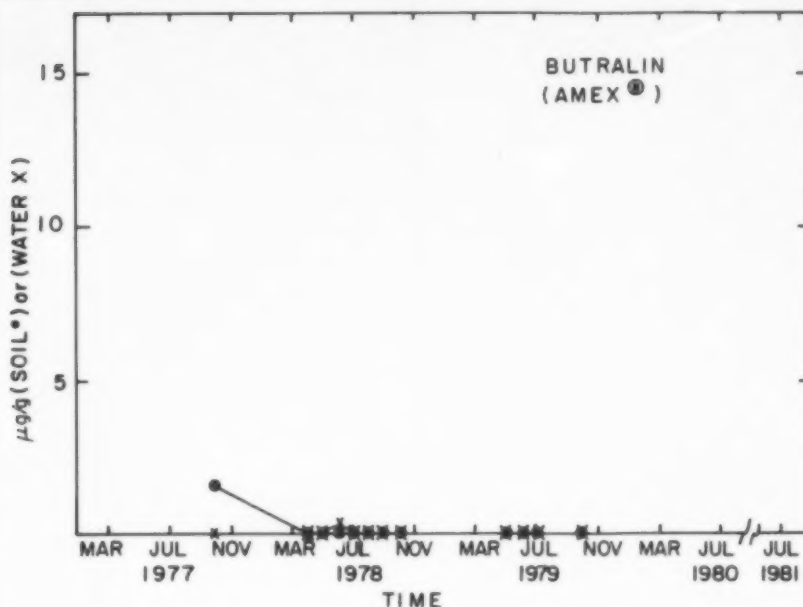


Figure 17. Butralin concentration in soil and water samples from horticulture macropit.

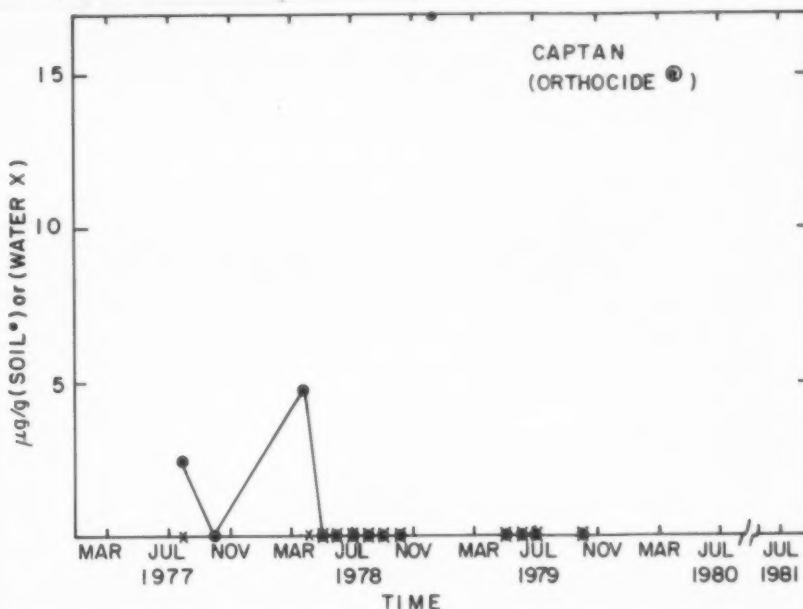


Figure 18. Captan concentration in soil and water samples from horticulture macropit.

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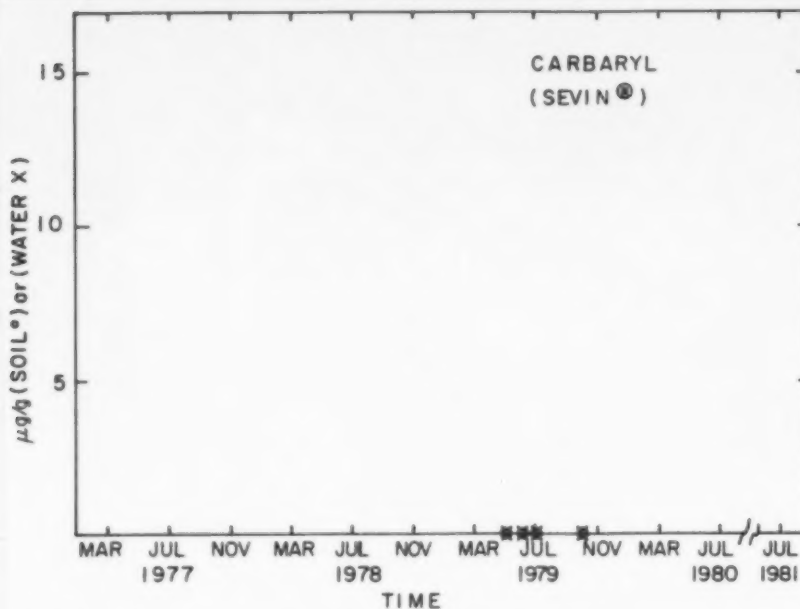


Figure 19. Carbaryl concentration in soil and water samples from horticulture macropit.

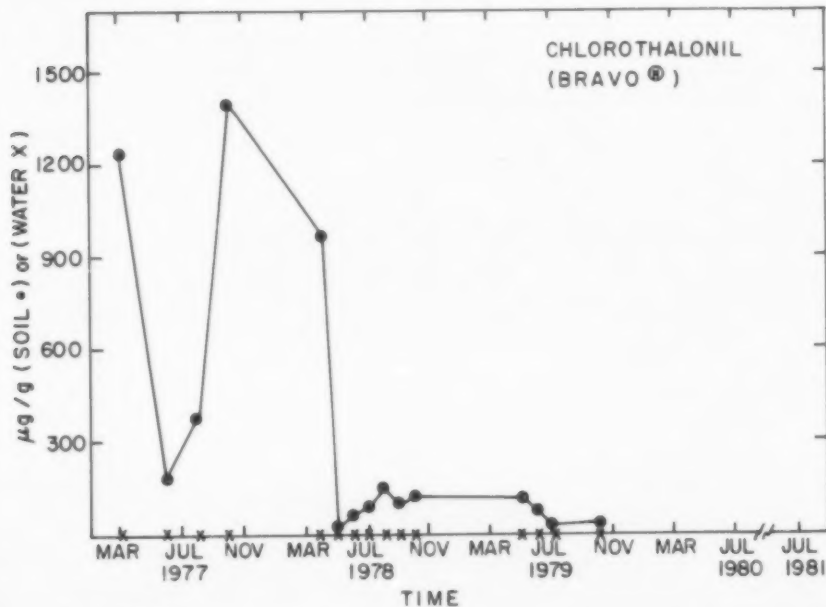


Figure 20. Chlorothalonil concentration in soil and water samples from horticulture macropit.

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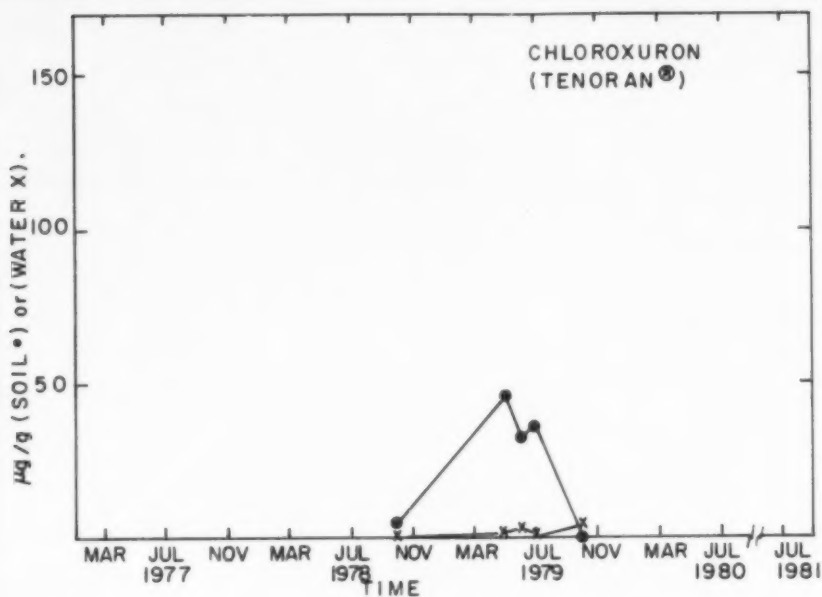


Figure 21. Chloroxuron concentration in soil and water samples from horticulture macropit.

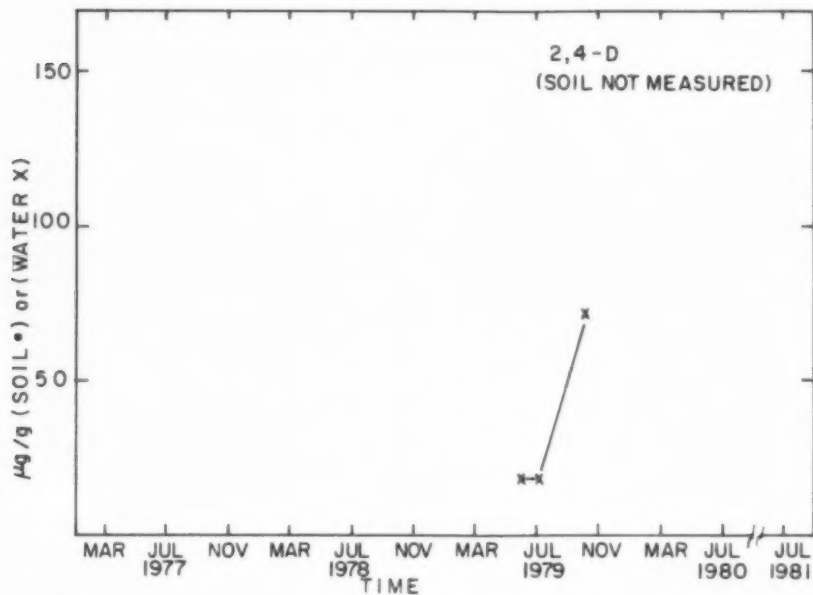


Figure 22. 2,4-D concentration in soil and water samples from horticulture macropit.

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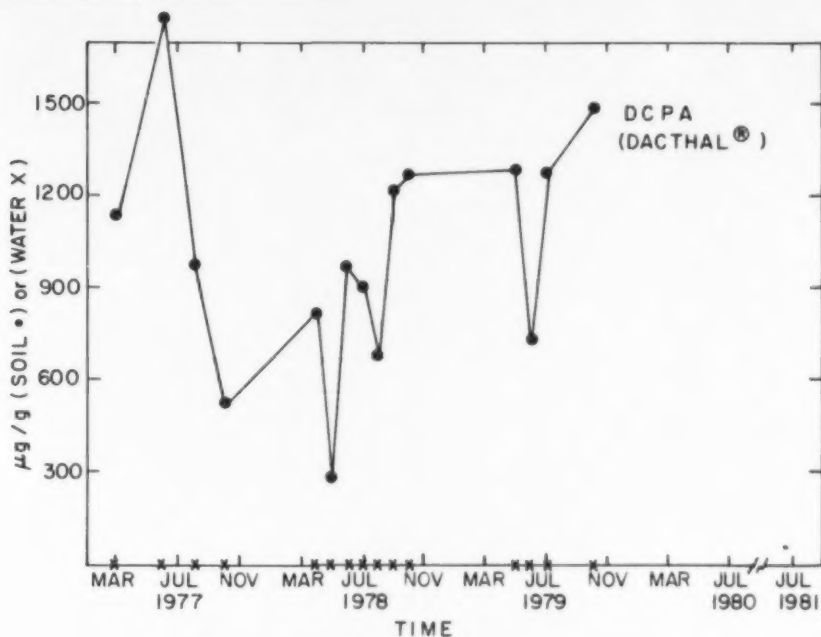


Figure 23. DCPA concentration in soil and water samples from horticulture macropit.

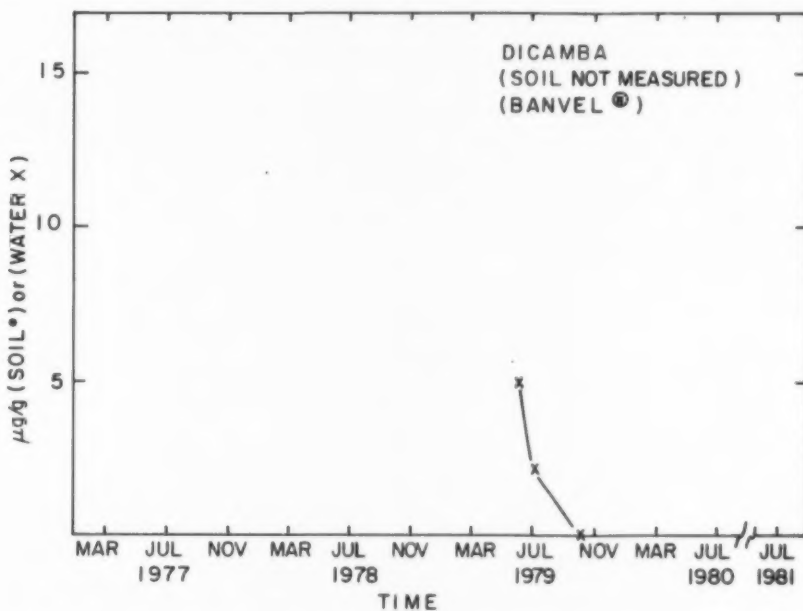


Figure 24. Dicamba concentration in soil and water samples from horticulture macropit.

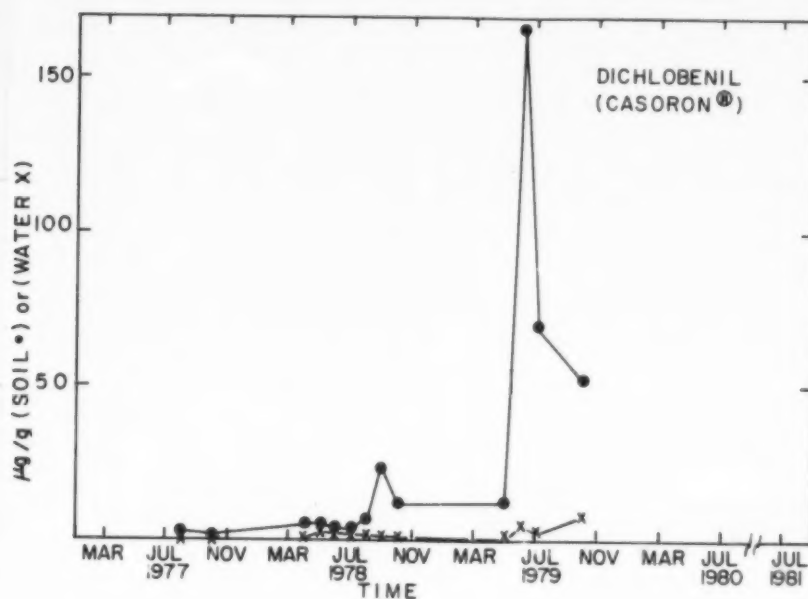
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Figure 25. Dichlobenil concentration in soil and water samples from horticulture macropit.

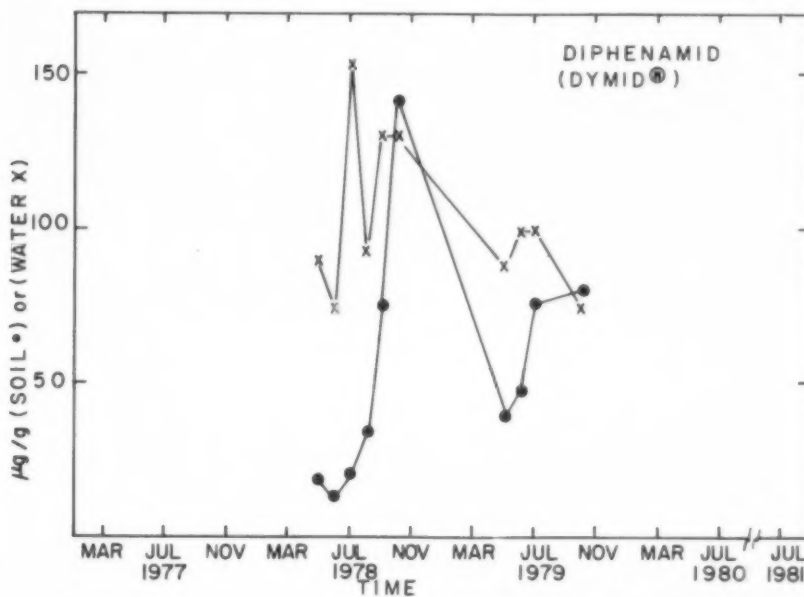


Figure 26. Diphenamid concentration in soil and water samples from horticulture macropit.

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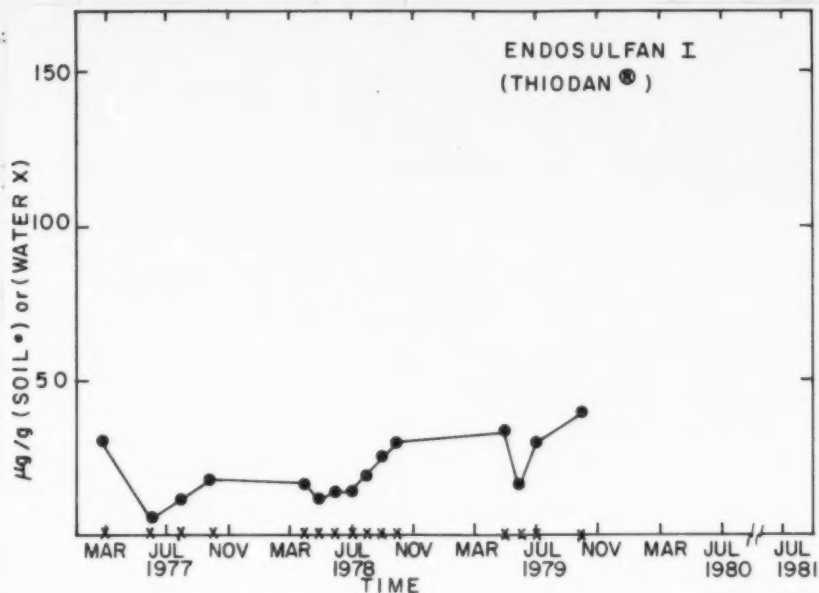


Figure 27. Endosulfan I concentration in soil and water samples from horticulture macropit.

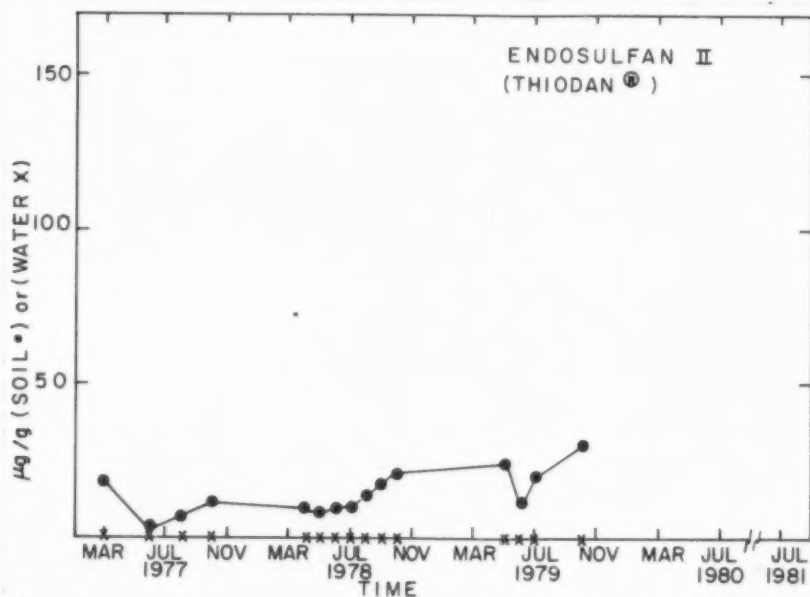


Figure 28. Endosulfan II concentration in soil and water samples from horticulture macropit.

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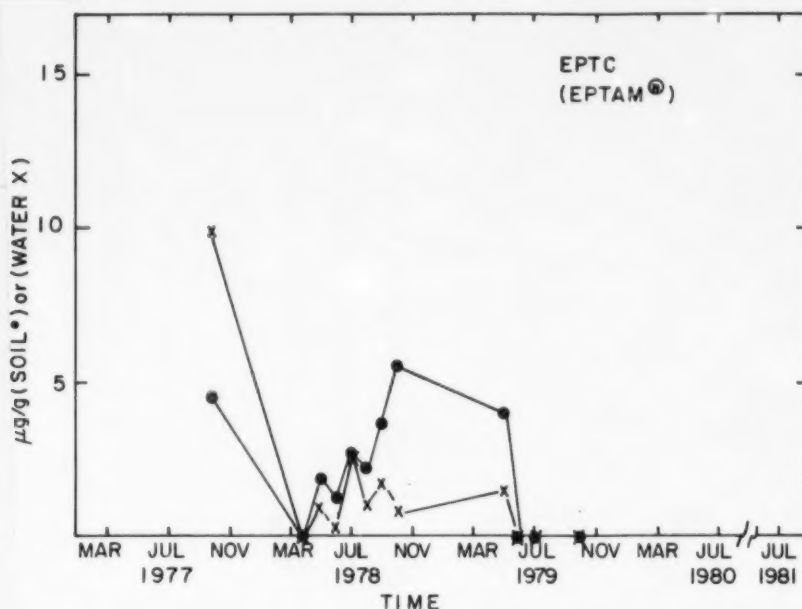


Figure 29. EPTC concentration in soil and water samples from horticulture macropit.

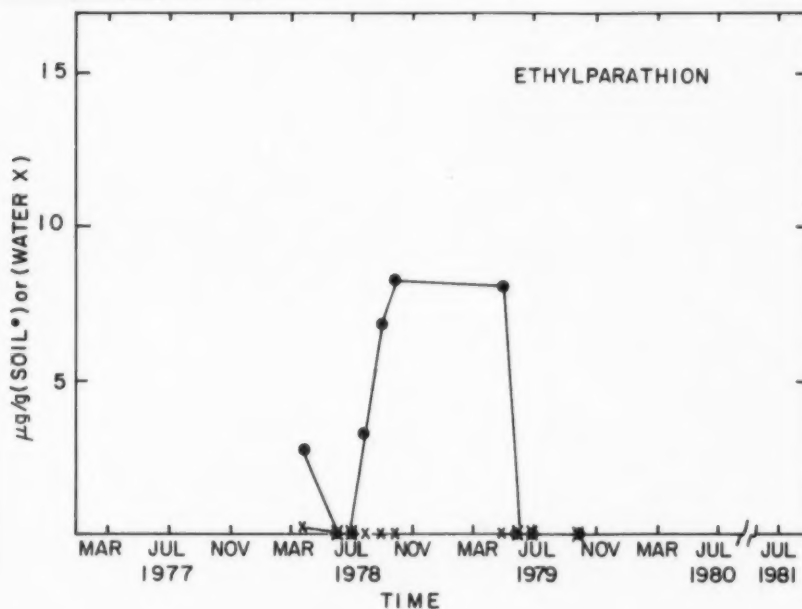


Figure 30. Ethylparathion concentration in soil and water samples from horticulture macropit.

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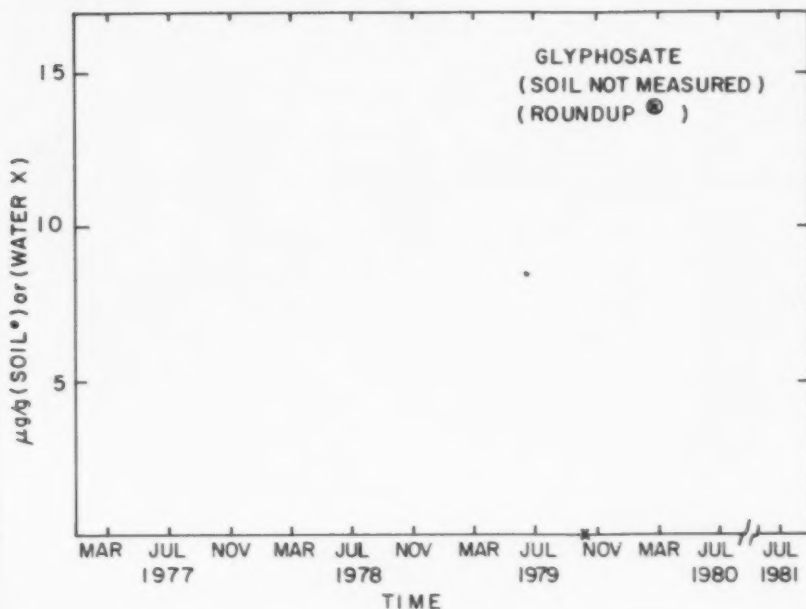


Figure 31. Glyphosate concentration in soil and water samples from horticulture macropit.

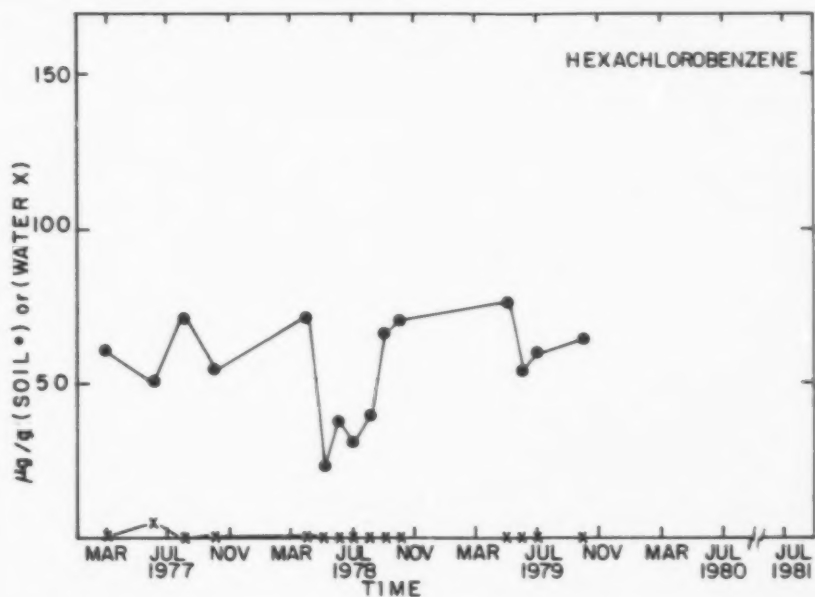


Figure 32. Hexachlorobenzene concentration in soil and water samples from horticulture macropit.

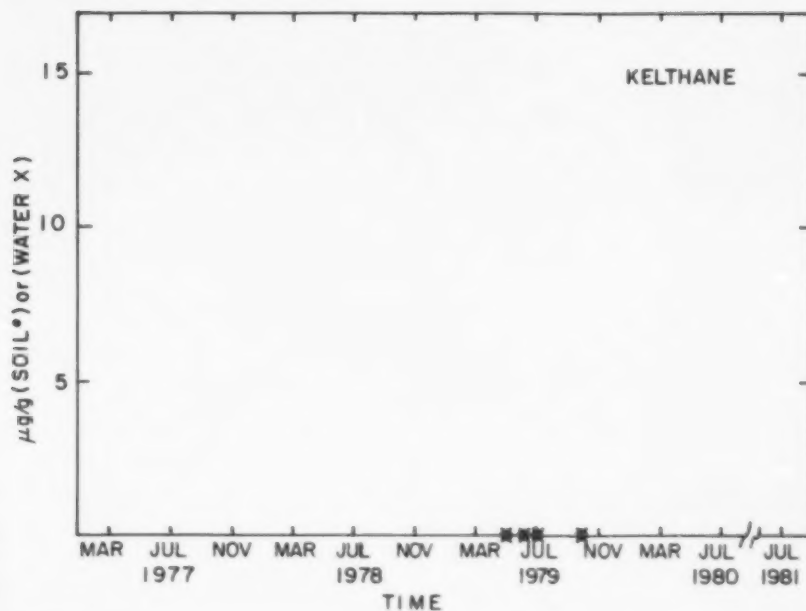


Figure 33. Kelthane concentration in soil and water samples from horticulture macropit.

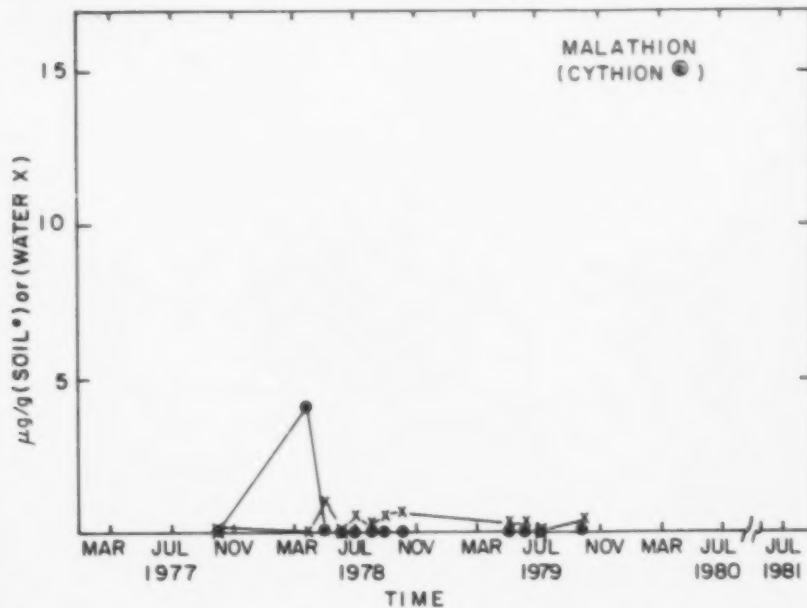


Figure 34. Malathion concentration in soil and water samples from horticulture macropit.

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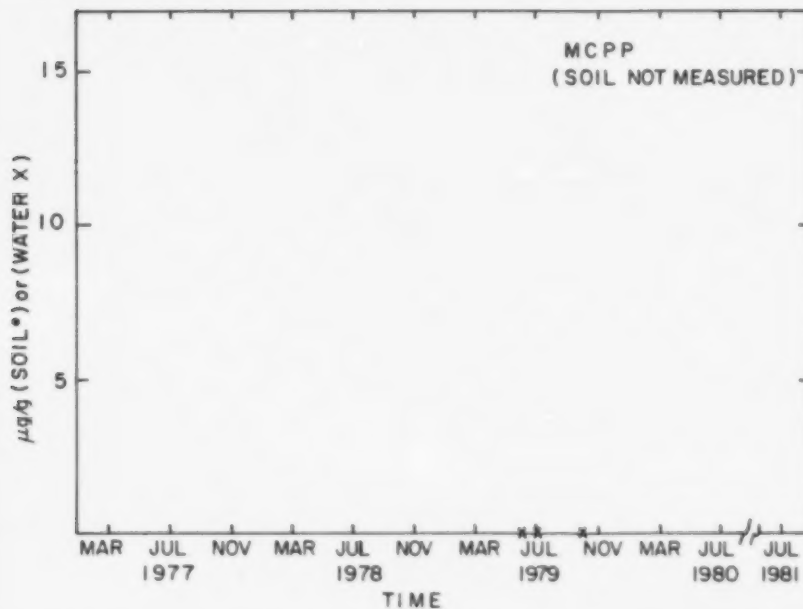


Figure 35. MCPP concentration in soil and water samples from horticulture macropit.

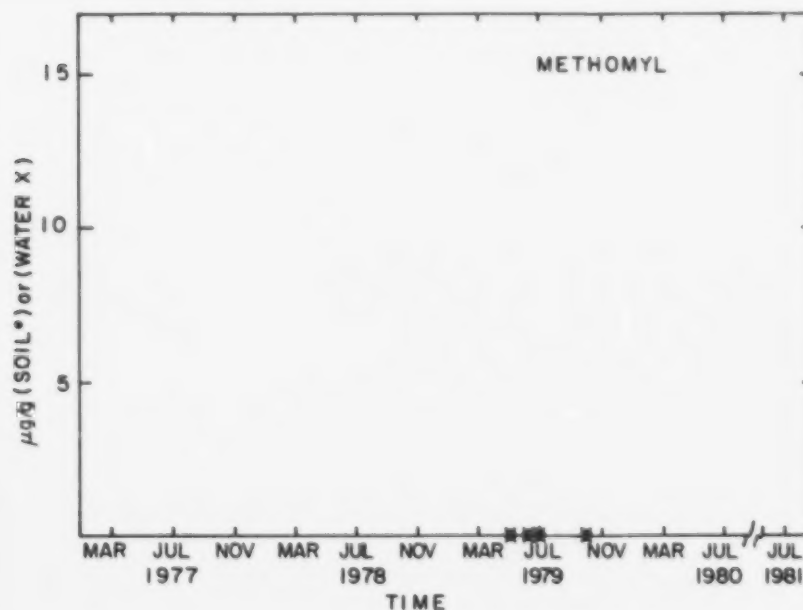


Figure 36. Methomyl concentration in soil and water samples from horticulture macropit.

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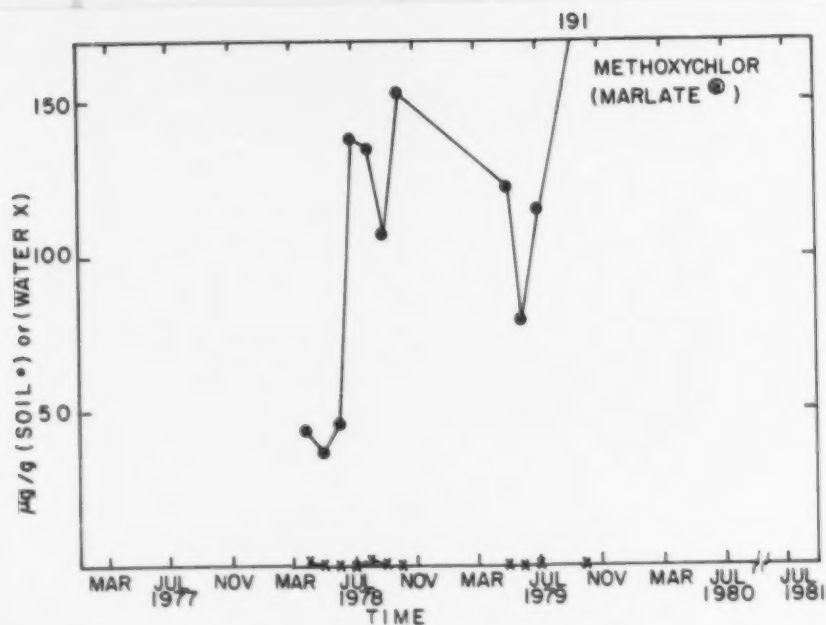


Figure 37. Methoxychlor concentration in soil and water samples from horticulture macropit.

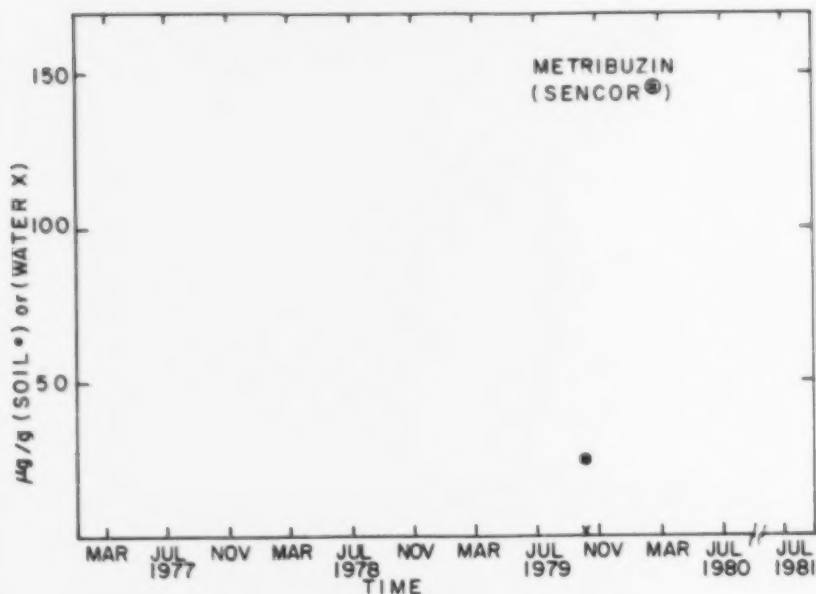


Figure 38. Metribuzin concentration in soil and water samples from horticulture macropit.

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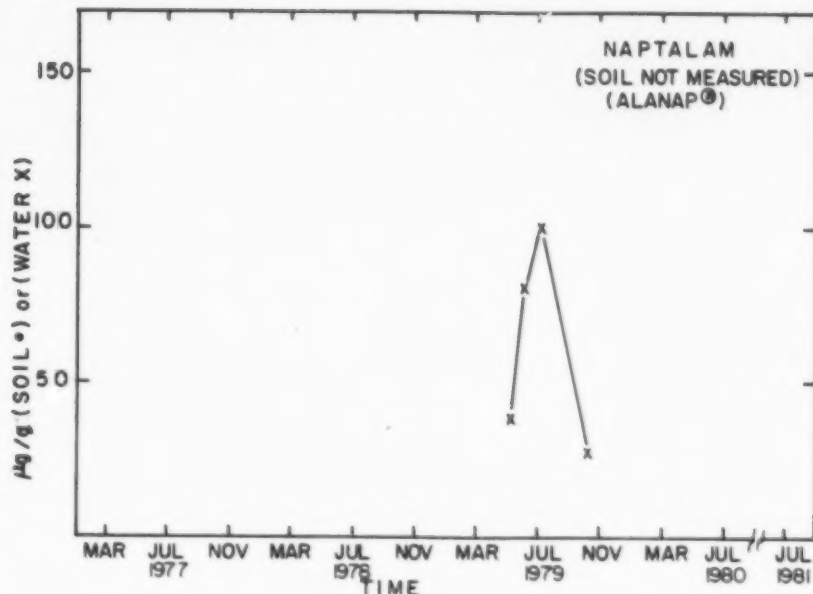


Figure 39. Naptalam concentration in soil and water samples from horticulture macropit.

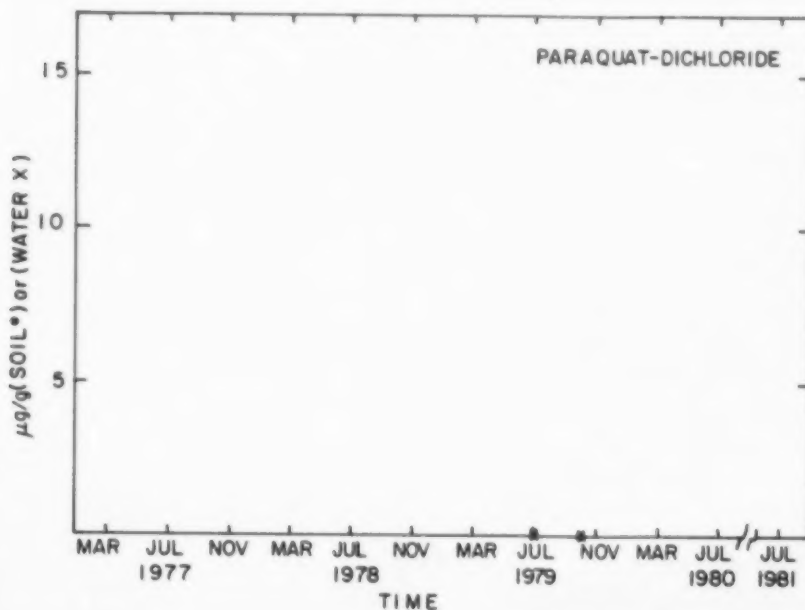


Figure 40. Paraquat concentration in soil and water samples from horticulture macropit.

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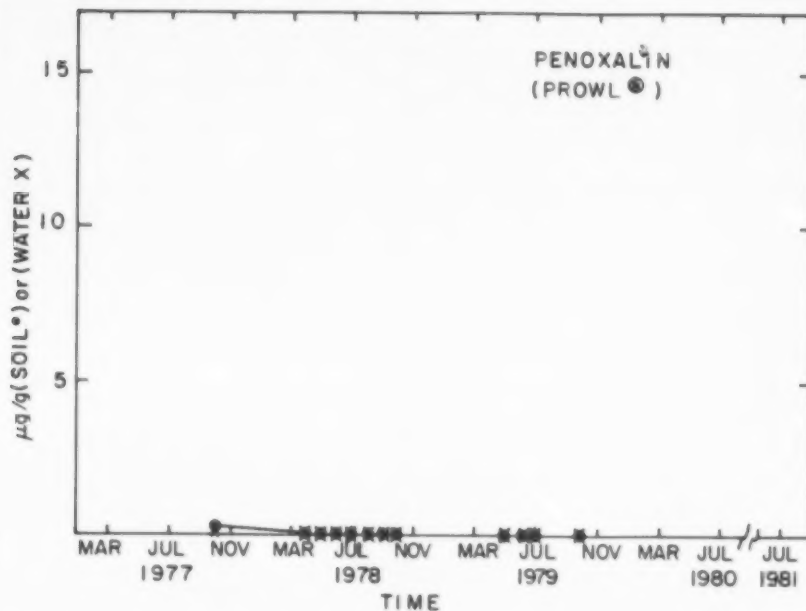


Figure 41. Penoxalin concentration in soil and water samples from horticulture macropit.

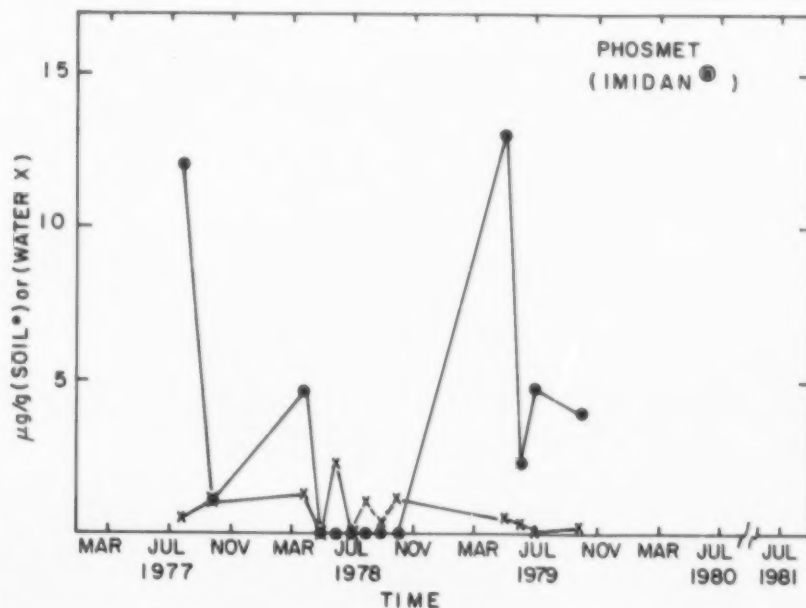


Figure 42. Phosmet concentration in soil and water samples from horticulture macropit.

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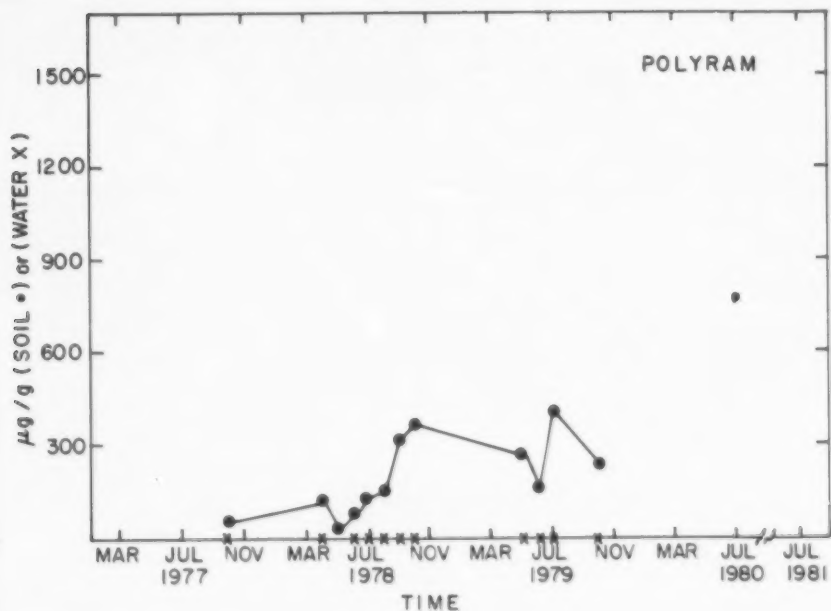


Figure 43. Polyram concentration in soil and water samples from horticulture macropit.

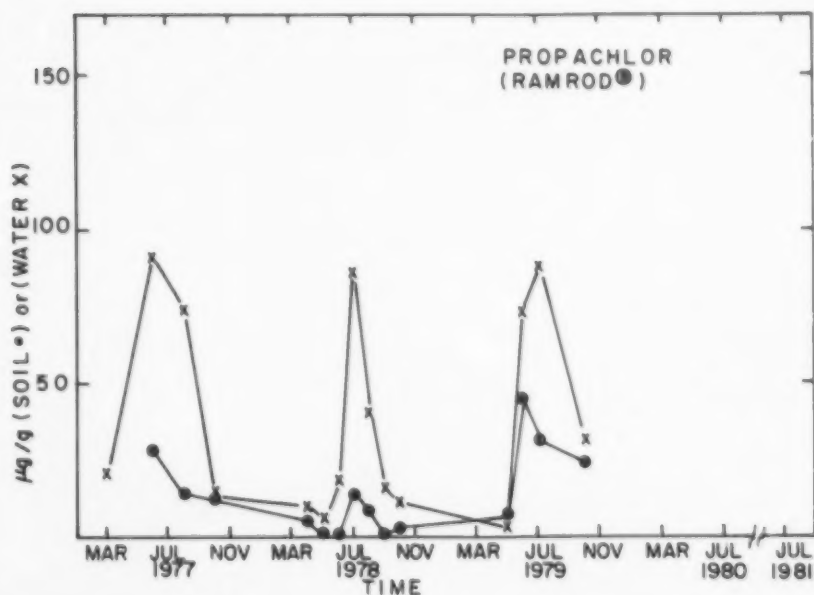


Figure 44. Propachlor concentration in soil and water samples from horticulture macropit.

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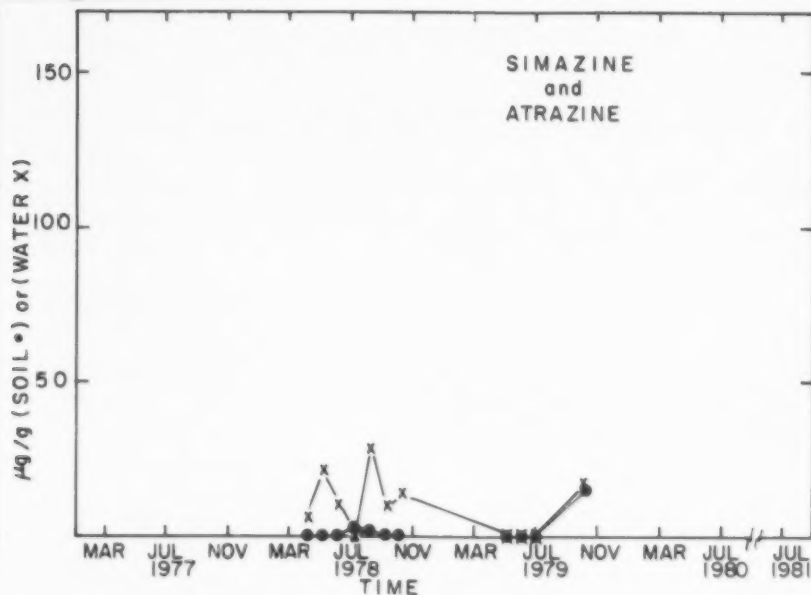


Figure 45. Simazine concentration in soil and water samples from horticulture macropit.

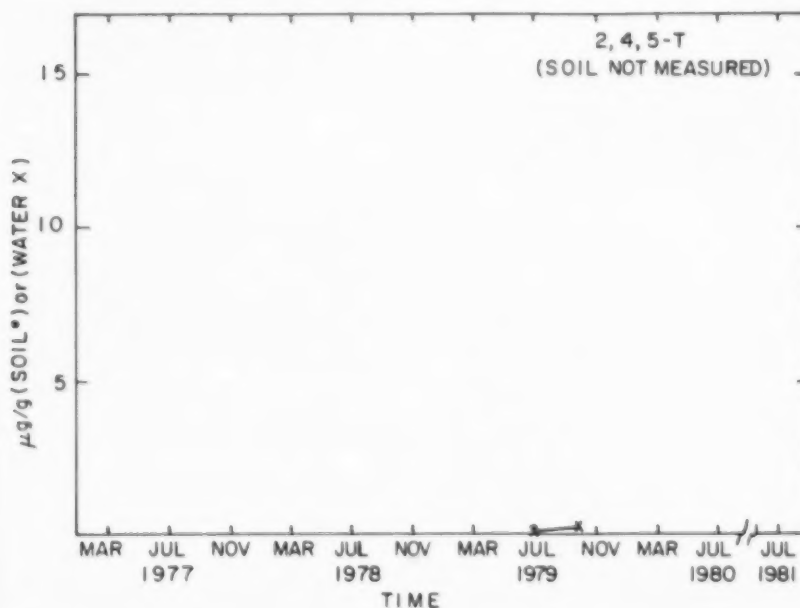


Figure 46. 2,4,5-T concentration in soil and water samples from horticulture macropit.

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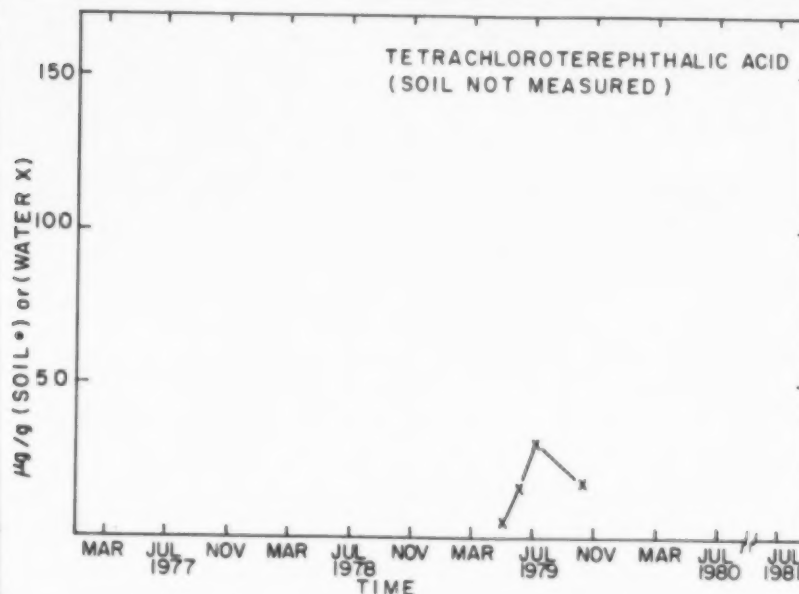


Figure 47. Tetrachloroterephthalic acid concentration in soil and water samples from horticulture macropit.

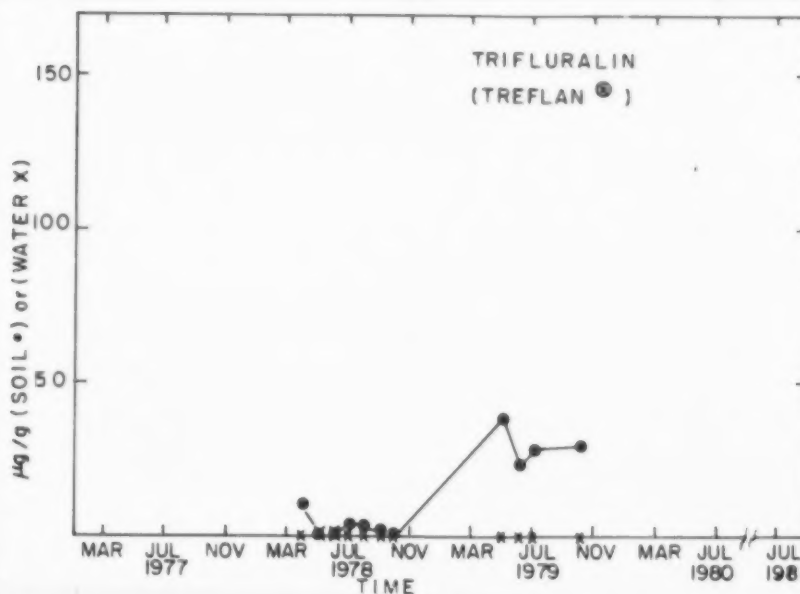


Figure 48. Trifluralin concentration in soil and water samples from horticulture macropit.

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Figures 13-48. Here the common names for the pesticides have been alphabetized for convenience of location. The formulations deposited are shown in parenthesis on the graphs. Originally these graphs were produced with the intention of conducting a monitor program over a period of five years to end in the summer of 1981. This explains the extended horizontal axes. The vertical axes are either 0-15, 0-150 or 0-1500 $\mu\text{g/g}$ soil or water depending on the maximum concentration observed for any one residue. In some cases, such as alachlor in Figure 13 and benomyl in Figure 15, no data points are shown because these pesticides were either not measured as is the case for alachlor in 1977 and 1978 or the analytical schemes had not yet been devised as is the case for benomyl in 1977. From some other pesticides such as dicamba shown in Figure 24, the 1977 and 1978 data points are absent because the pesticide had not yet been dumped into the pit.

All data points in Figures 13-48 and later on in Figures 49-68 are connected by straight lines. The graphical data thus give a quick visual representation of probable build-up only. It is not a representation of decay per se. Probable decay or loss of pesticides due to incomplete containment may be deduced by careful inspection of these data and the deposition data given in Table 13. However the massage of the analytical data by mathematical adjustments of the concentrations based on deposition factors is unsuccessful for deducing degradation because of the random nature of the sampling and deposition dates and the unknown losses due to decay, incomplete containment and irreversible adsorptions. The analytical uncertainties, due to the highly heterogeneous nature of these pits, also argue against mas-saging the analytical data with deposition factors in order to deduce whether degradation has occurred.

In general most of the pesticide residues are immobilized on soil particles, although in some cases, such as alachlor, atrazine, benomyl, EPTC, diphenamid, naptalam and propachlor, amounts comparable to that found on the soil were found in the liquid phase. This presence in the liquid phase, which is primarily aqueous, can be very significant if the liquid contents are inadvertantly lost due to leakage. This consideration is discussed in more detail in the section on Containment of Pesticides.

Horticulture Micropits

Four small pits, called micropits, were constructed in the Spring of 1977 and positioned adjacent to the macropit at the Horticulture farm. The construction details for these micropits can be found elsewhere in this report.

The entire history of use of these micropits could be documented more fully than the use of the macropit. Whenever a deposition was made into the macropit, a portion of the dump was also deposited into the appropriate micropit if the pesticide could be classified as applicable to vegetable, fruit or ornamental use. These deposition records are reported elsewhere in this report. No really useful information was accumulated from these efforts with the micropits.

Soil and Liquid Analyses--

The results of the water and soil analyses of samples taken from the vegetable, fruit and ornamental micropits are given in Tables 15, 16 and 17 respectively. A fourth micropit, used as a control, was also sampled and analyzed but no tabular data are presented here since the pesticides were not present at the detection limits.

The attempt to gain added insights into the degradations and possible build-up of undesirable chemicals using data from these more controlled micropits was not successful. The results from analyses of samples from these micropits were not any more enlightening than the results for the macropit because of the uncontrolled variables discussed in the preceeding section and the additional variable of an unknown adaptation factor for the microorganisms responsible for the degradations. Another complicating factor contributing to the failure of these micropit systems was the loss of the liquid contents during the winter and spring of 1979 when ruptures of the encased polyethylene containers occurred. Following the detection of these ruptures in the Spring of 1979, soil samples were taken from below the vegetable and fruit micropits. These were analyzed and the results are tabulated in Table 18. These data show no dramatic contamination of the surrounding soil due to the ruptures but any conclusions from interpretation of these data alone are highly questionable because of: 1) sampling uncertainties; 2) possible loss of some solid soil contents from the suspended containers; 3) possible build-up from vapor phase movement of pesticides from the macropit; and 4) inadvertant contamination of the soil during any one or all of the pesticide depositions into these micropits.

Surroundings - Horticulture Station

Water samples were taken from a tile system immediately surrounding the macropit and from sites more remote from the location of the disposal pit. Air samples were taken from both above and upwind from the disposal pit. The air samples were analyzed to arrive at an estimate of the pesticides lost due to evaporation. The water samples were analyzed to ascertain leakage and to determine if this seven year old disposal pit had contaminated the surrounding ground water to a measurable extent over that which occurs from other probable sources.

The water and air analyses results are discussed separately in the following sections. From interpretation of these results very little contamination of either the surrounding ground water or the ambient air can be attributed to the operation of this particular pit system.

Water Analyses--

The results of the analyses of water samples taken adjacent to and remote from the macro disposal pit are given in Table 19. The data under the columns labelled adjacent to pit represents the amounts of the pesticides found in the tile immediately surrounding the pit. Any large amount of a soluble pesticide observed here would be indicative of leakage from the pit and a first order indicator of possible contamination of surrounding ground

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TABLE 15 PESTICIDES IN SOIL AND WATER AT HORTICULTURE FARM MICRO DISPOSAL PIT - VEGETABLE

Pesticide	in	Amount in PPM													
		Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978**	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	May 29 1979	Jun 28 1979	Sep 21 1979	
Benomyl	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	12	0.2 4.1	0.8 13	
Bensulide (Prefar)	H ₂ O Soil	-	-	-	-	-	-	*	-	-	-	-	*	1.4 74	
Carbaryl (Sevin)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	*	
Chlorothalonil (Bravo)	H ₂ O Soil	*	<0.1 9.1	0.5 *	0.5 -	0.2 *	* *	2.0 1.2	0.5 32.8	0.7 *	0.4 108	- 371	<0.1 76	0.3 65	
2,4-D	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	*	
DCPA (Dacthal)	H ₂ O Soil	<0.1 *	<0.1 1.1	0.1 *	<0.1 -	<0.1 448	0.7 7.9	8.2 10.8	0.8 92.8	0.6 63.4	0.4 329	- 595	0.2 125	0.4 66	
Dicamba	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	*	
Diphenamid (Dymid)	H ₂ O Soil	-	-	-	-	*	*	34.7 *	13.9 10.8	86.2 *	69.3 51.4	- 22	2.0 3.1	4.0 *	
Endosulfan I (Thiodan)	H ₂ O Soil	0.2 *	0.1 5.8	0.3 7.9	0.2 -	0.2 *	0.4 22.3	0.9 7.4	0.9 33.3	0.3 10.9	0.2 43.1	- 75	<0.1 24.9	0.1 28	
Endosulfan II (Thiodan)	H ₂ O Soil	*	<0.1 2.7	0.1 4.0	<0.1 -	<0.1 0.2	0.3 12.9	0.6 4.2	0.4 20.8	0.2 6.1	0.1 26.6	- 56	<0.1 16.2	0.2 19	
EPTC (Eptam)	H ₂ O Soil	-	-	-	-	-	-	-	0.1 0.3	*	*	- *	*	*	
Hexachlorobenzene	H ₂ O Soil	<0.1 *	*	<0.1 *	0.1 -	* 22.1	* 0.1	* *	* 0.2	* 0.1	* 0.5	- 0.6	* 0.2	* 0.2	

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Amount in ppm

Pesticide	In	Jun 2 1977	Aug 2 1977	Oct 6 1977	Apr 6 1978**	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	May 29 1979	Jun 28 1979	Sep 21 1979
Kelthane	H ₂ O Soil	*	*	*	-	*	*	*	*	*	*	-	*	*
Malathion (Cythion)	H ₂ O Soil	*	*	*	<0.1	0.3	*	0.4	*	*	*	-	*	*
MCPP	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	*
Methomyl	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	*	*
Methoxychlor (Marlate)	H ₂ O Soil	*	*	*	<0.1	<0.1	<0.1	1.8	1.6	0.4	0.1	-	<0.1	<0.1
Metribuzin (Sencor)	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	0.7
Polyram	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	2.9	1.4
Pr. pachlor (Ramrod)	H ₂ O Soil	52 0.9	9.0 2.9	*	0.3	<0.1	0.4	121 9.2	73 19.3	24.1 5.7	0.5 7.3	-	*	*
Simazine (Princep)	H ₂ O Soil	*	*	*	*	5.4	*	*	97.1 5.2	4.4	8.4	-	*	11
Tetrachloro- terephthalic Acid	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	34
Trifluralin (Treflan)	H ₂ O Soil	*	*	*	*	0.1	*	*	*	*	*	-	<0.1	0.2

★★ Frozen-no soil sample taken

- Not measured

TABLE 16 PESTICIDES IN SOIL AND WATER AT HORTICULTURE FARM MICRO DISPOSAL PIT - FRUIT

Pesticide	in	Amount in PPM													
		Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978**	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	May 29 1979	Jun 28 1979	Sep 21 1979	
Alachlor (Lasso)	H ₂ O Soil	*	*	*	*	*	*	*	*	*	*	*	*	*	
Azinphos-Methyl (Guthion)	H ₂ O Soil	*	<0.1 80	0.9 15.7	*	<0.1 *	*	*	*	*	*	*	*	*	
Benomyl	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	0.2 2.7	0.3 5.0	
Butralin (Amex)	H ₂ O Soil	2.2 *	*	*	*	*	*	*	*	*	*	*	*	*	
Captan	H ₂ O Soil	<0.1 *	<0.1 *	*	*	*	*	*	*	*	*	*	*	*	
Chloroxuron	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	21	*	
DCPA (Dacthal)	H ₂ O Soil	0.6 65	0.8 508	0.7 1231	0.9 -	0.4 1.3	0.5 343	2.9 705	0.6 810	0.4 386	0.3 1081	-	0.2 324	0.1 424	
Dichlobenil (Casoron)	H ₂ O Soil	1.3 12	*	<0.1 4.6	<0.1 -	2.2 0.6	0.6 2.2	0.2 3.6	<0.1 3.6	<0.1 3.2	<0.1 5.5	-	<0.1 1.7	<0.1 8	
Diphenamid (Dymid)	H ₂ O Soil	-	*	*	*	30.4 *	9.6 *	60.7 *	34.6 13	14.7 14.5	45.7 20	-	*	*	
Endosulfan I (Thiodan)	H ₂ O Soil	*	<0.1 *	*	<0.1 -	<0.1 25.8	<0.1 *	*	<0.1 *	<0.1 0.2	<0.1 0.7	-	*	<0.1 0.4	
Endosulfan II (Thiodan)	H ₂ O Soil	*	*	*	<0.1 -	<0.1 15.1	<0.1 *	0.3	<0.1 0.6	<0.1 0.4	<0.1 1.3	-	*	<0.1 0.6	

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TABLE 16 (continued)

Pesticide	in	Amount in PPM													
		Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978**	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	May 29 1979	Jun 28 1979	Sep 21 1979	
Hexachlorobenzene	H ₂ O Soil	<0.1 21	*	<0.1 58.7	<0.1 -	<0.1 *	<0.1 7.6	*	20.8	<0.1 12.4	<0.1 30	-	<0.1 15	<0.1 25	
Methoxychlor (Mariate)	H ₂ O Soil	*	*	*	*	<0.1 3.6	0.2 *	0.3 2.4	<0.1 9.6	<0.1 14.4	<0.1 26.2	-	<0.1 19	*	
Permethrin (Procl)	H ₂ O Soil	*	*	*	<0.1 -	*	*	*	*	*	*	*	*	*	
Phosmet (Imidan)	H ₂ O Soil	<0.1 1.9	*	*	*	*	<0.1 *	*	*	*	*	*	*	*	
Polyram	H ₂ O Soil	-	-	-	-	*	3.6 22.2	22.4 81.1	10.5 51	7.7 50	9.3 118	-	4.7 25	*	
Tetrachloro- terephthalic Acid	H ₂ O Soil	-	-	-	-	-	-	-	-	-	-	-	-	105 -	

* Not detected

** Frozen-no soil sample taken

- Not measured

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TABLE 17 PESTICIDES IN SOIL AND WATER AT HORTICULTURE FARM MICRO DISPOSAL PIT - ORNAMENTAL

Pesticide	in	Amount in PPM												
		Jun 2 1977	Aug 2 1977	Oct 4 1977	Apr 4 1978**	May 1 1978	May 31 1978	Jun 27 1978	Jul 26 1978	Aug 29 1978	Sep 27 1978	Ma/ 28 1979	Jun 28 1979	Sep 21 1979
2,4-D	H ₂ O	-	-	-	-	-	-	-	-	-	-	-	-	*
	Soil	-	-	-	-	-	-	-	-	-	-	-	-	*
DCPA (Dacthal)	H ₂ O	<0.1	*	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Soil	0.9	1.8	*	-	0.6	0.9	*	0.4	*	0.2	*	*	*
Dicamba	H ₂ O	-	-	-	-	-	-	-	-	-	-	-	-	*
	Soil	-	-	-	-	-	-	-	-	-	-	-	-	-
Dichlobenil (Casoron)	H ₂ O	<0.1	*	*	*	<0.1	<0.1	*	*	*	*	*	*	*
	Soil	*	*	*	-	*	*	*	*	*	*	*	*	*
Hexachlorobenzene	H ₂ O	*	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Soil	*	1.3	7.1	-	11.4	47.9	10.1	9.3	5.2	10.8	7.3	7.1	4
MCPP	H ₂ O	-	-	-	-	-	-	-	-	-	-	-	-	*
	Soil	-	-	-	-	-	-	-	-	-	-	-	-	-
Tetrachloro- terephthalic Acid	H ₂ O	-	-	-	-	-	-	-	-	-	-	-	-	19.1
	Soil	-	-	-	-	-	-	-	-	-	-	-	-	-

* Not detected

** Frozen-no soil sample taken

- Not measured

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TABLE 18. AMOUNTS OF PESTICIDES IN PPM IN SOIL SAMPLES FROM BELOW
MICRO DISPOSAL PITS AFTER RUPTURES DETECTED IN SPRING 1979

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Pesticides	PPM	
	Vegetable	Fruit
Benomyl	0.5	1.2
Chlorothalonil (Bravo)	0.01	
Chloroxuron (Tenoran)		0.06
DCPA (Dacthal)	0.13	0.06
Dichlobenil (Casoron)		0.02
Diphenamid (Dymid)		0.51
Endosulfan I (Thiodan)	0.07	0.002
Endosulfan II (Thiodan)	0.03	0.003
Hexachlorobenzene	0.01	0.01
Methoxychlor (Marlate)	0.04	0.02
Propachlor (Ramrod)	0.01	
Trifluralin (Treflan)	0.01	

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TABLE 19. PESTICIDES IN WATER SAMPLES TAKEN AT THE HORTICULTURE FARM

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Pesticide	Amount in PPM (mg/l)					
	Adjacent to Pit			Hort. Pond.		Various†
	Apr 1978	Jun 1979	Sep 1979	Apr 1978	Jun 1979	Jul 1978
Alachlor	*	*	*	*	0.001	0.0017
Bensulide	-	*	*	-	-	-
Butralin	*	*	*	*	-	-
Captan	*	*	*	*	*	-
Carbaryl	-	*	-	-	-	-
Chlorothalonil	*	0.020	*	0.0009	*	*
Chloroxuron	*	*	*	*	*	-
2,4-D	-	0.006	0.010	-	0.0015	-
2,4-DB	-	*	*	-	*	-
DCPA†	0.0016	0.002	0.0036	0.004	0.0009	0.0016
Dicamba	-	0.0006	*	-	0.0002	-
Dichlobenil	*	0.010	*	*	*	*
Diphenamid	*	*	*	*	*	-
Endosulfan I	0.001	*	*	0.0001	*	*
Endosulfan II	*	*	*	*	*	*
EPTC	*	*	*	*	*	-
Ethyl parathion	*	*	*	*	*	*
Folpet	*	*	*	*	*	-
Hexachlorobenzene	*	*	*	0.0001	*	*
Kelthane	*	*	*	*	*	*
Malathion	*	*	*	*	*	*

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Pesticide	Amount in PPM (mg/l)					
	Adjacent to Pit			Hort. Pond.		Various [†]
	Apr 1978	Jun 1979	Sep 1979	Apr 1978	Jun 1979	Jul 1978
MCPP	-	*	*	-	*	-
Methomyl	-	*	-	-	-	-
Methoxychlor	0.015	*	*	*	*	*
Naptalam	-	*	*	-	*	-
Penoxalin	*	*	*	*	*	-
Phosmet	0.015	*	*	*	*	*
Propachlor	*	*	0.060	0.005	0.0001	*
Simazine + Atrazine	*	*	0.050	*	0.007	*
Trifluralin	0.010	0.040	*	*	*	*
2,4,5-T	-	0.0003	*	-	*	-
Tetrachloroterephthalic Acid	-	0.041	0.330	-	0.005	-

*Not detected

-Not measured

[†] Average concentration of samples collected at twelve remote locations at the Horticulture Farm.

[‡] DCPA measured Aug. 1978 and Apr. 1979 was 0.001 and 0.006 PPM respectively in the water adjacent to the pit.

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water. Repeat analyses over a relatively long time period are necessary for proper interpretation of these data since inadvertant contamination may distort the analyses for any short period of time. Nevertheless, the periodic analyses of the ground water from the channel surrounding the macropit are useful as a first indicator of a problem in the operation of the disposal pit.

The Horticulture pond designated in Table 19 is located about 1000 yards from the disposal site and it receives seepage and run-off from all surrounding areas. Water from this pond has measurable amounts of several pesticides but this contamination is related to run-off from surrounding farm land rather than contamination from the macropit. The chemicals and the amounts observed here in the early spring of 1978 and 1979 are typical for surface water from any farm pond in Central Iowa. The undetectable pesticide amounts in the samples taken in July 1978 at the various remote locations on the farm are also typical of drainage water from any Iowa farm in July. The data are not related to the amounts of the pesticides deposited in the disposal pit.

Water was also taken from a well located within 50 yards of the disposal pits and no pesticides were found at the detection limits of 1 to 10 ng/l which is about 10^5 times lower than the detection limit for the samples reported in Table 19.

Air Analyses--

The results of the analyses of the air taken above and upwind from the disposal pit are given in Tables 20 and 21 for the spring and fall of 1979. The amounts observed are very low except for propachlor in the spring of 1979. This relatively high value may be caused by an uneven dump of propachlor just prior to the sampling date at a position close to the location of the sampler.

Contamination from the pit may be assumed whenever there is an appreciable increase in pesticide concentration for air taken over the pit relative to the upwind sample taken at the same time. The penoxalin data for the June 28, 1979 samples are anomalous. This anomaly may be due to an unknown point source and unusual wind conditions.

Sufficient samples at the proper locations were not taken to allow for accurate estimates of the vaporization losses for those pesticides which could be proven to be emanating from the disposal pit. However average wind conditions and the amounts measured can be used to calculate maximum losses by evaporation. These losses are not great and the limited amounts of pesticides which are evaporated from the pit are diluted rapidly in the atmosphere. The pit therefore is a source of air contamination due to incomplete containment but the amount of contamination is negligible.

Agronomy Macropits

The accumulations of the amounts of pesticides deposited in the Agron-

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TABLE 20. AMOUNTS OF PESTICIDES IN ng/l AIR COLLECTED AT
HORTICULTURE DISPOSAL PIT ON JUNE 28, 1979

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Pesticide	Up-Wind From Pit	On Top ‡ of Pit
DCPA	<0.1	1.1
Dichlobenil	<0.1	2.5
Endosulfan I	*	0.3
Endosulfan II	*	<0.1
Hexachlorobenzene	*	0.3
Methoxychlor	*	<0.1
Penoxalin	1.0†	<0.1
Propachlor	*	7.5†
Treflan	*	*
2,4-D (Acid)	*	*
2,4,5-T (Acid)	*	*

TABLE 21. AMOUNTS OF PESTICIDES IN ng/l AIR COLLECTED AT
HORTICULTURE DISPOSAL PIT ON SEPT. 21, 1979

Pesticide	Up-Wind From Pit	On Top ‡ of Pit
DCPA	<0.1	1.2
Dichlobenil	0.2	1.1
Endosulfan I	*	0.2
Endosulfan II	*	<0.1
Hexachlorobenzene	*	0.3
Methoxychlor	*	0.1
Penoxalin	0.2†	0.1
Propachlor	*	0.5†
Treflan	*	0.5
2,4-D (Acid)	*	*
2,4,5-T (Acid)	*	*

‡ Downwind corner of pit

* Not detected

† Tentative identification based only on coincidence of t_R

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omy macropit #2 are given in Table 22. This macropit was constructed in the late fall of 1977 and first used in February of 1978. It is a much newer system than the Horticulture macropit and the thirteen year old Agronomy macropit #1. The use of macropit #1 was abandoned in 1977 and no samples were taken for chemical analyses because of the difficulty of getting a uniform sample from the highly compacted soil and gravel contents of the pit and because of the very uncertain records of chemical depositions. Plant assays described elsewhere in this report were chosen for estimating the extent of residual herbicides in this disposal pit. However ground water samples in the vicinity of both macropits #1 and #2 were taken to provide an indication of ground water contamination due to leakage from either or both pit systems.

The design of the macropit #2 precluded getting soil samples from the pit so only liquid samples were taken for analyses. Even though data already discussed for the Horticulture macropit and data to be discussed later on for the minipits show that most of the pesticides are present on soil particles it is not necessary to measure the amount immobilized on the soil in order to make an assessment of the utility of a pit disposal system since loss of the solid contents is highly improbable short of a catastrophic earthquake. The liquid contents can be lost through cracks and ruptures but data from the minipit study prove that the soil within the pit acts like a sorption column to effectively capture even the highly soluble pesticides before the liquid, which is mostly water, seeps through to the soil surrounding the pit. However there is always the possibility that the soil in the pit might have very low or zero capacity for some pesticides. Therefore the amounts of pesticides in the liquid phase should be measured regularly to account for losses due to leakage and to estimate the maximum contamination from the most adverse leakage problem. In practice, the rupture should be repaired before any significant escape of pesticides occurs due to leakage of the liquid contents. Since some small amount of all pesticides are present in the liquid phase, the regular sampling and analyses of this phase is also desirable for estimating whether a build-up, which can be detrimental, is occurring. The interpretation of the monitor data also provides first order evidence of degradation.

The Agronomy macropit #2 is another uncontrolled disposal system so the analytical data are perturbed by the same variables of amount, timing, decay and losses as described in the discussion of the Horticulture macropit.

Liquid Analyses--

The amounts of the pesticide residues in the liquid samples taken from the macropit #2 at the Agronomy farm are tabulated in Table 23. The numerical data represent the average of five separate analyses of samples taken from five sampling tiles for the April 31 and June 21, 1978 dates. All other entries are for samples from the five tiles which were composited prior to extraction and analysis. As noted for the Horticulture macropit, none of pesticides deposited in this Agronomy pit seem to be building up in concentration. This is as expected because of the immobilization and degradation capacity of the soil contents of the pit system.

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The data in Table 23 are better represented by the graphical plots in

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TABLE 22. DISPOSAL ACCUMULATIONS AT THE AGRONOMY MACRO PIT #2
IN GRAMS OF ACTIVE INGREDIENT (AI)*

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Pesticide Deposited	Grams AI at Listed Dates	
	Nov 1, 1978	Oct 1, 1978
Aatrex	17488	21393
Amiben	1898	2511
Banvel	45	272
Basagran	1249	2612
Bexton	1380	7987
Bladex	6973	13452
Cygon	123	212
2,4-D amine	558	1175
Dual	1021	1171
Eradicane	1807	3668
Furloe	2561	5853
Lasso	31031	53634
Malathion	427	427
Paraquat	300	800
Prowl	227	272
Ramrod	11186	11186
Ravage	45	45
Roundup	4087	5086
Sencor	3722	4675
Sevin	†	204
Surflan	45	45
Sutan	4654	4745
Tolban	91	91
Treflan	10138	11409
TOTAL	= 101056	152925

* 4540 g of an unspecified number of pesticides were also dumped on or about Feb 1, 1978. The total grams and the listings are thus absolute minimums.

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† Entry made in log book but no amounts listed.

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TABLE 23. HERBICIDES IN LIQUID SAMPLES FROM THE
AGRONOMY MACRO DISPOSAL PIT #2

Herbicide	Amount in PPM								
	Apr31 1978	Jun21 1978	Jul20 1978	Aug29 1978	Sep27 1978	May8 1979	May29 1979	Jun28 1979	Aug14 1979
Alachlor (Lasso)	1606	2023	1113	318	374	200	767	213	171
Amiben (Chloramben)	-†	-	-	-	-	-	*	*	*
Atrazine (Aatrex)	123	64	311	279	14	135	53	51	42
Basagran ‡ (Bentazon)	-	-	-	-	-	-	126	175	120
Butylate (Sutan)	112	112	49	13	9	*	15	4	3
Chlorpropham (Furloe)	160	136	51	15	12	13	28	20	5
Cyanazine (Bladex)	224	298	243	97	166	97	168	60	56
2,4-D ‡	-	-	-	-	-	-	10	39	37
Dicamba ‡ (Banvel)	-	-	-	-	-	-	-	15	5
Dimethoate (Cygon)	9	3	*	1	*	*	*	*	*
Diphenamid (Dymid)	-	-	-	-	-	*	*	*	*
EPTC (Eptam)	47	40	19	7	7	194	18	7	6
Glyphosate (Roundup)	-	-	-	-	-	-	*	*	*
Malathion (Cythion)	*	*	*	*	*	*	*	*	*
Metolachlor (Dual)	*	*	*	*	*	*	*	*	*
Metribuzin (Sencor)	-	-	203	110	-	111	50	156	106
Paraquat- dichloride	-	-	-	-	-	-	-	22	14
Profluralin (Tolban)	*	*	*	*	*	*	*	*	*
Propachlor (Ramrod)	41	293	189	132	153	99	145	171	63
2,4,5-T ‡	-	-	-	-	-	-	2	117	3
Trifluralin (Treflan)	129	1734	398	189	118	9	59	65	41

*Not detected

†Not measured

‡As methyl ester

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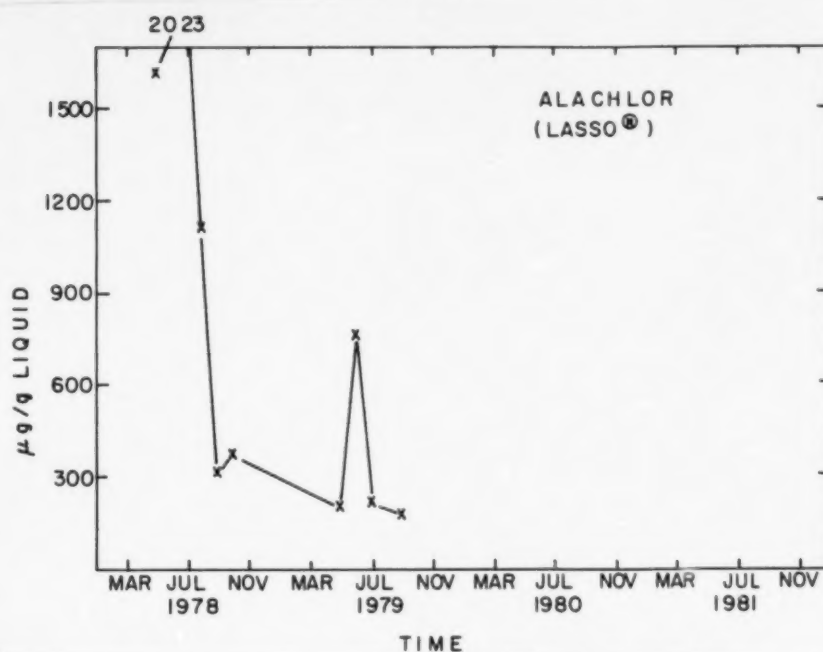
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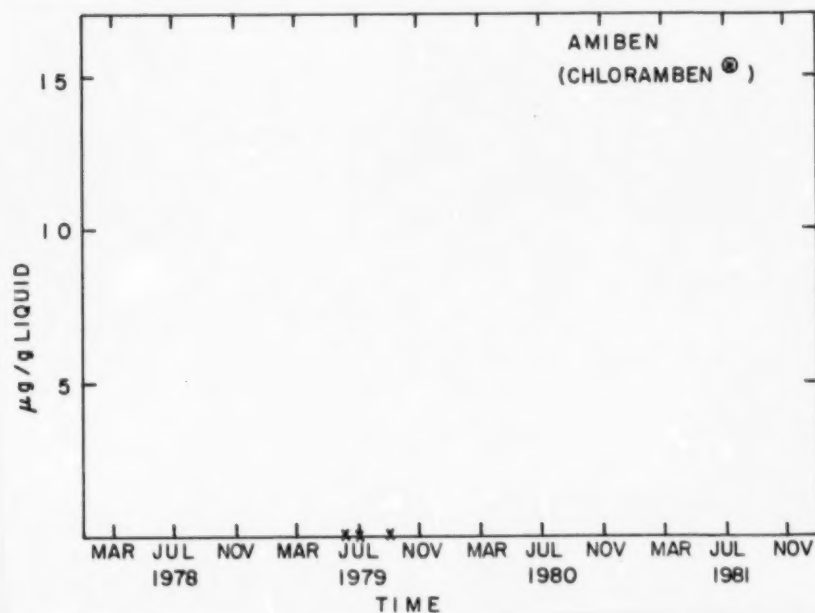
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4 Figure 49. Alachlor concentration in liquid samples from agronomy macropit #2.



5 Figure 50. Amiben concentration in liquid samples from agronomy macropit #2.

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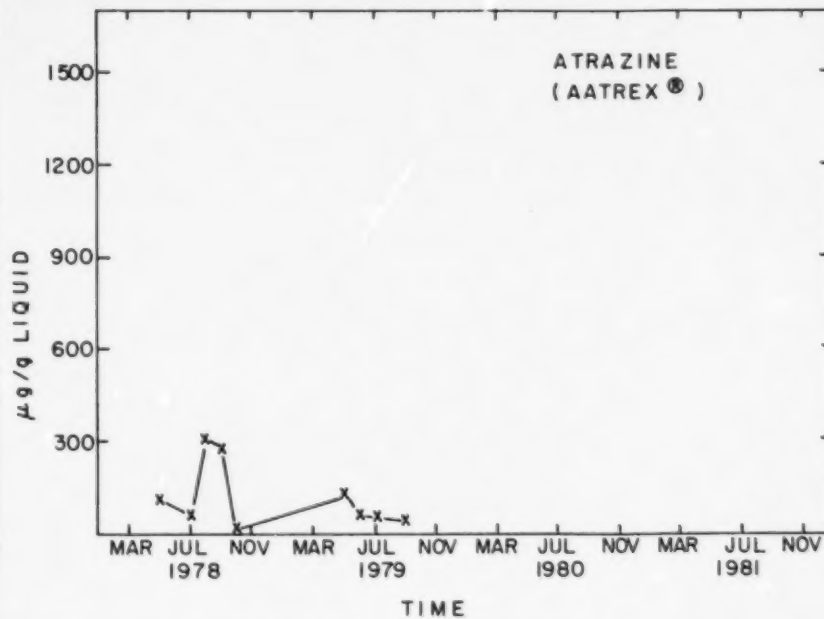
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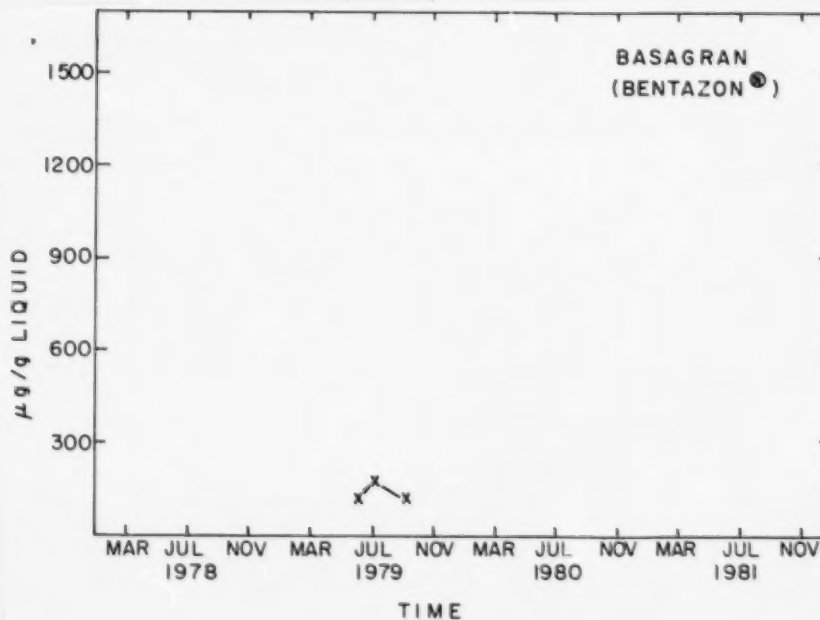
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51 Figure 51. Atrazine concentration in liquid samples from agronomy macropit #2.



52 Figure 52. Basagran concentration in liquid samples from agronomy macropit #2.

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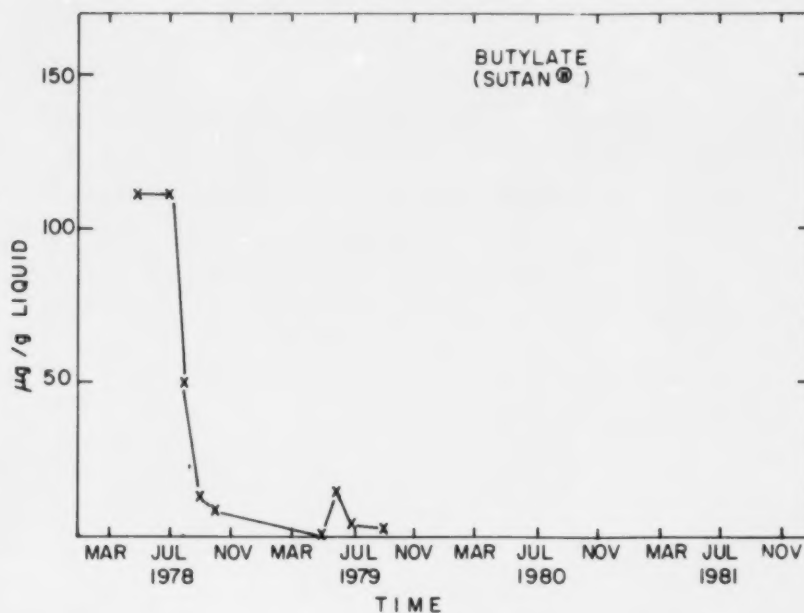


Figure 53. Butylate concentration in liquid samples from agronomy macropit #2.

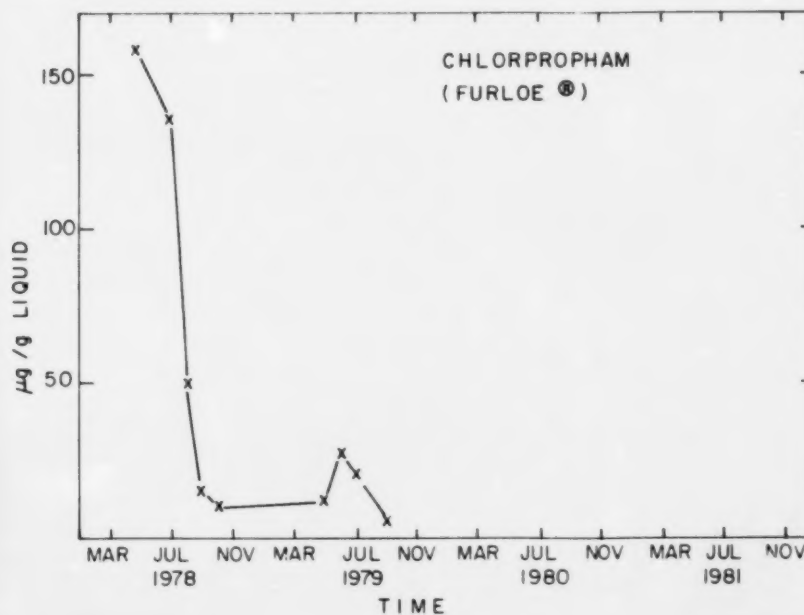


Figure 54. Chlorpropham concentration in liquid samples from agronomy macropit #2.

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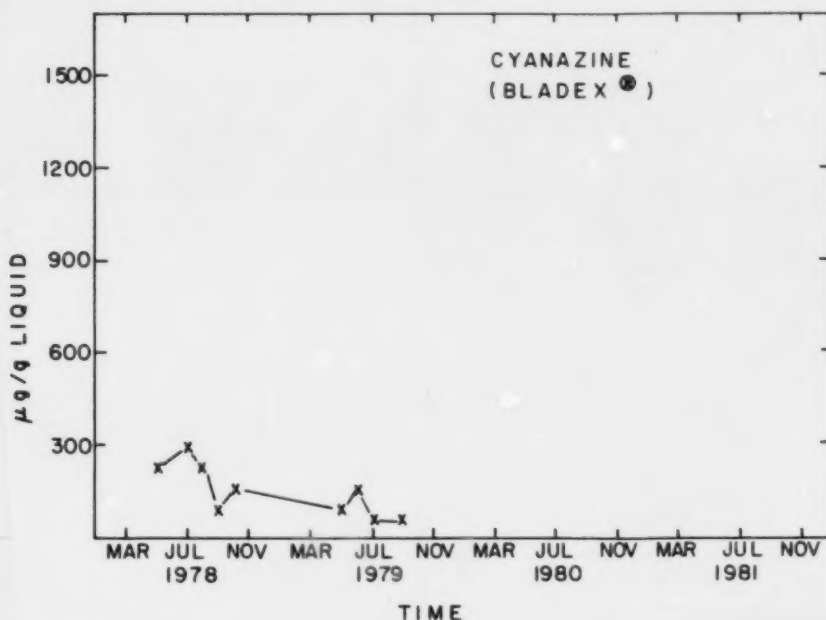
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55 Figure 55. Cyanazine concentration in liquid samples from agronomy macropit #2.

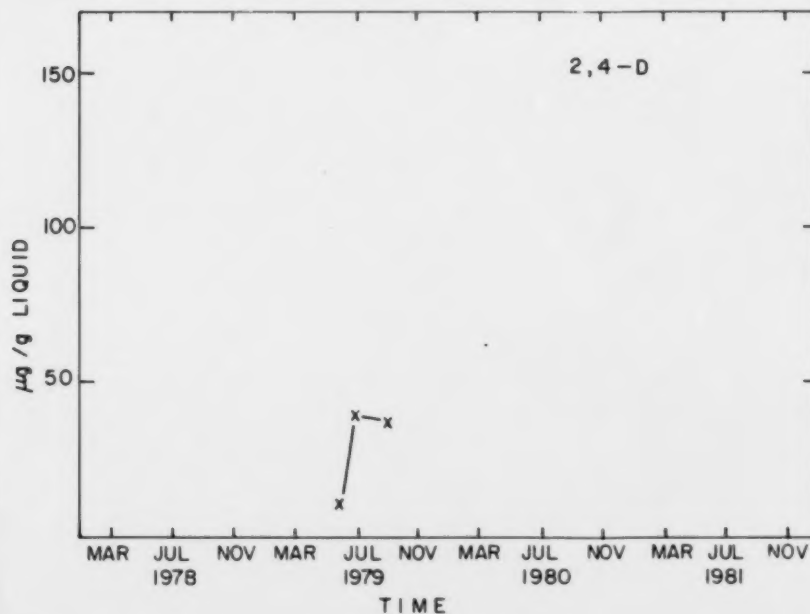


Figure 56. 2,4-D concentration in liquid samples from agronomy macropit #2.

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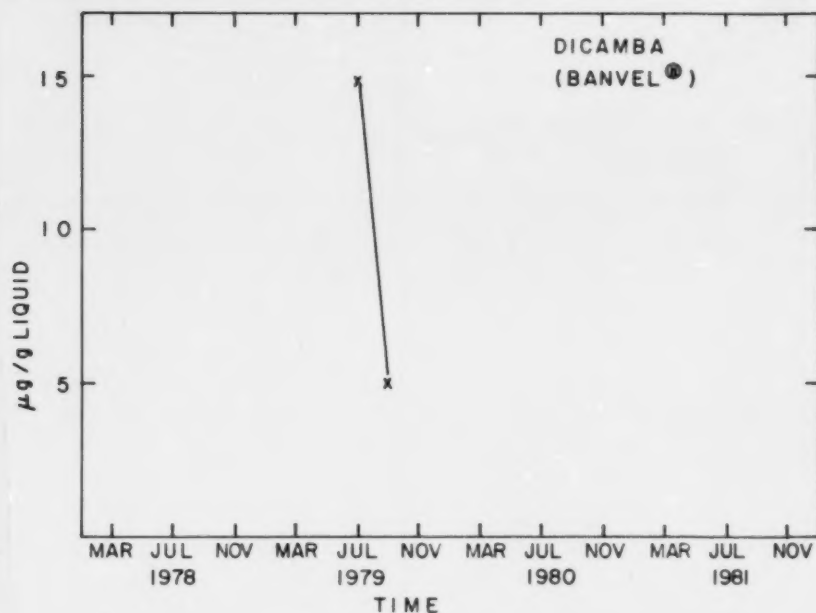


Figure 57. Dicamba concentration in liquid samples from agronomy macropit #2.

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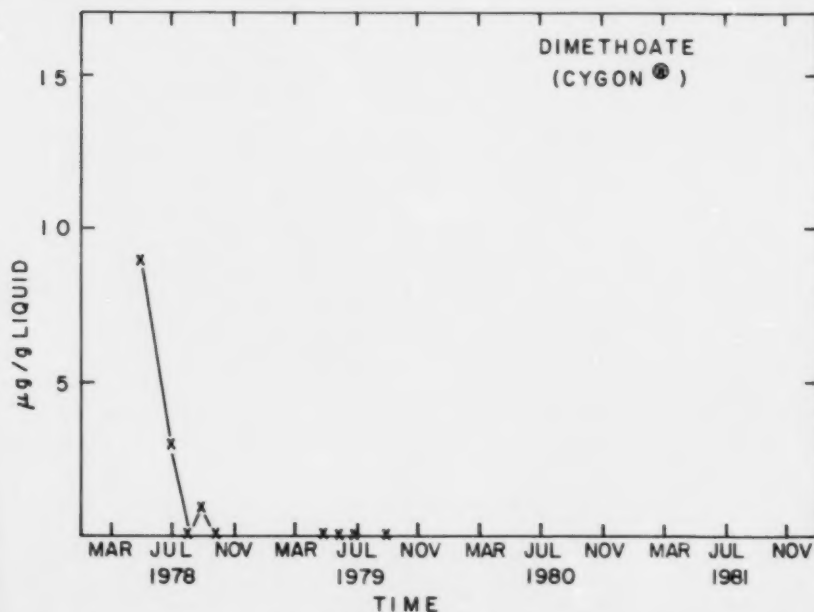


Figure 58. Dimethoate concentration in liquid samples from agronomy macropit #2.

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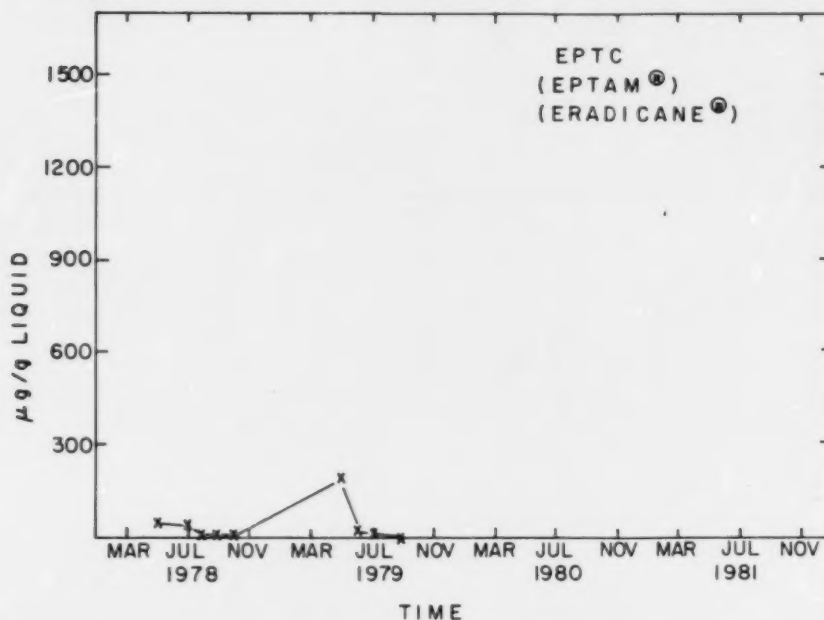
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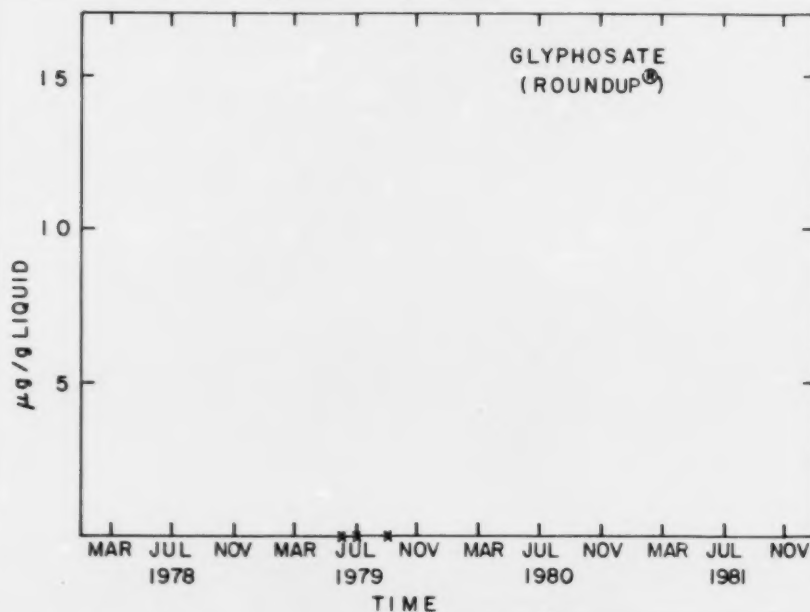
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5 Figure 59. EPTC concentration in liquid samples from agronomy macropit #2.



6 Figure 60. Glyphosate concentration in liquid samples from agronomy macropit #2.

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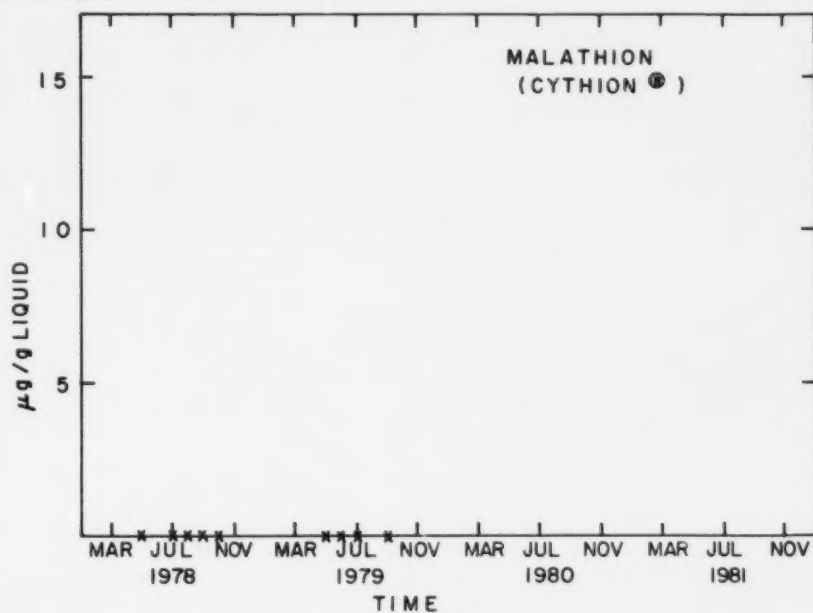


Figure 61. Malathion concentration in liquid samples from agronomy macropit #2.

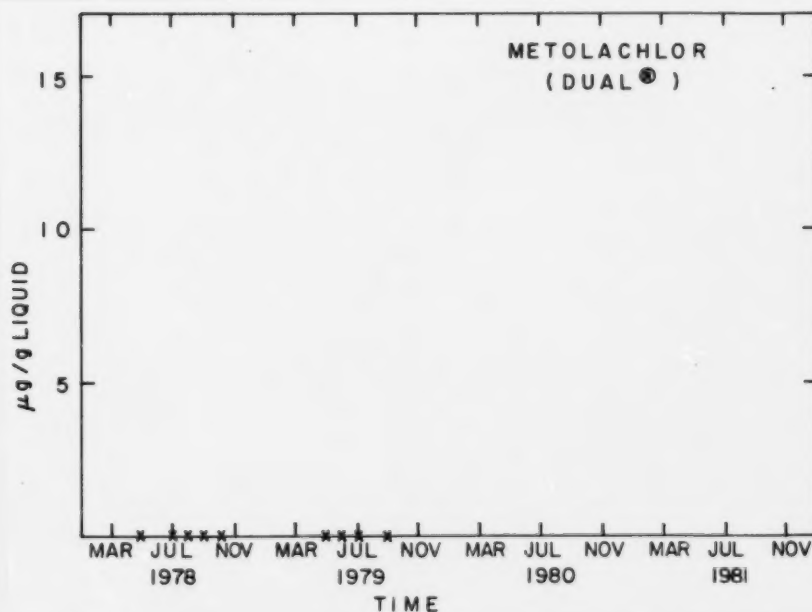


Figure 62. Metolachlor concentration in liquid samples from agronomy macropit #2.

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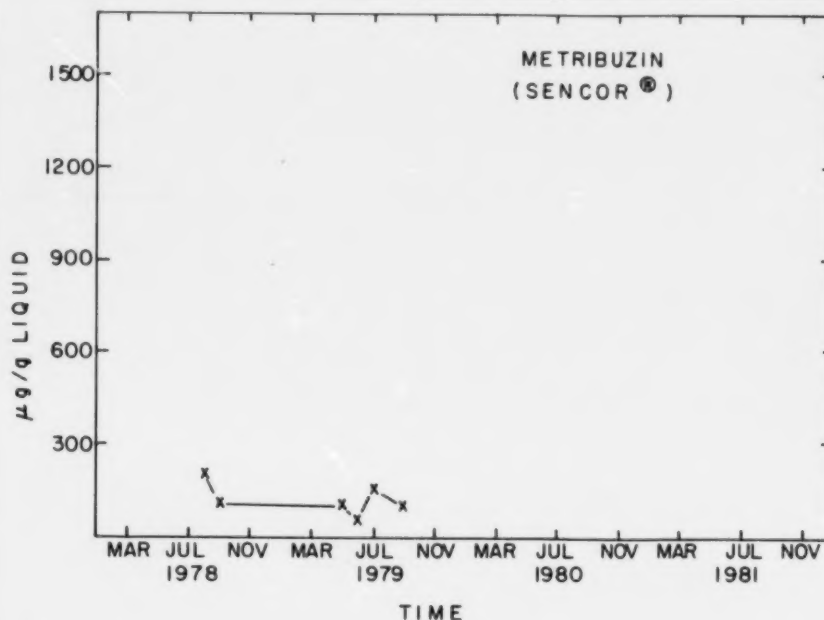


Figure 63. Metribuzin concentration in liquid samples from agronomy macropit #2.

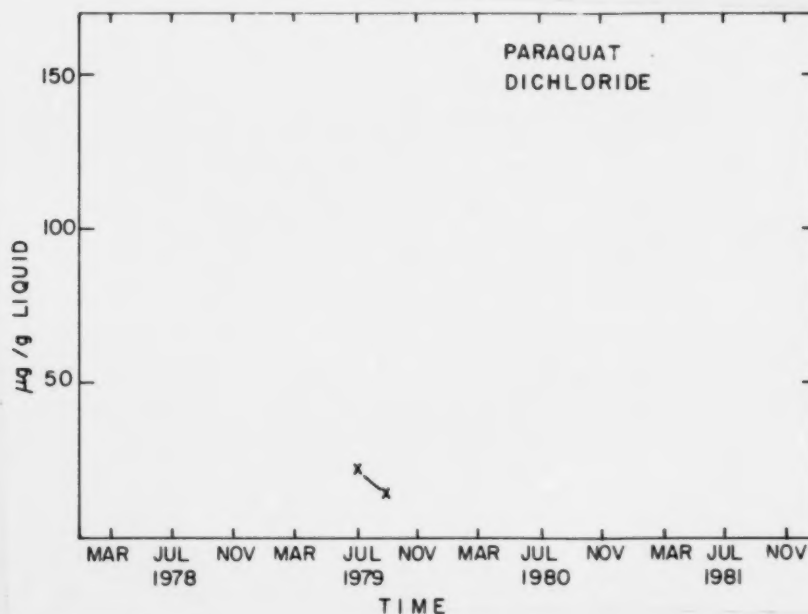


Figure 64. Paraquat concentration in liquid samples from agronomy macropit #2.

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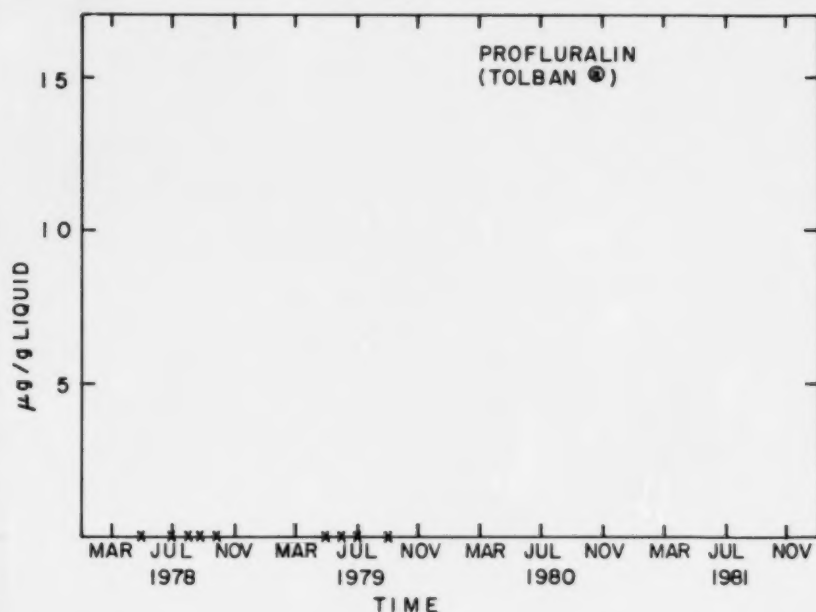


Figure 65. Profluralin concentration in liquid samples from agronomy macropit #2.

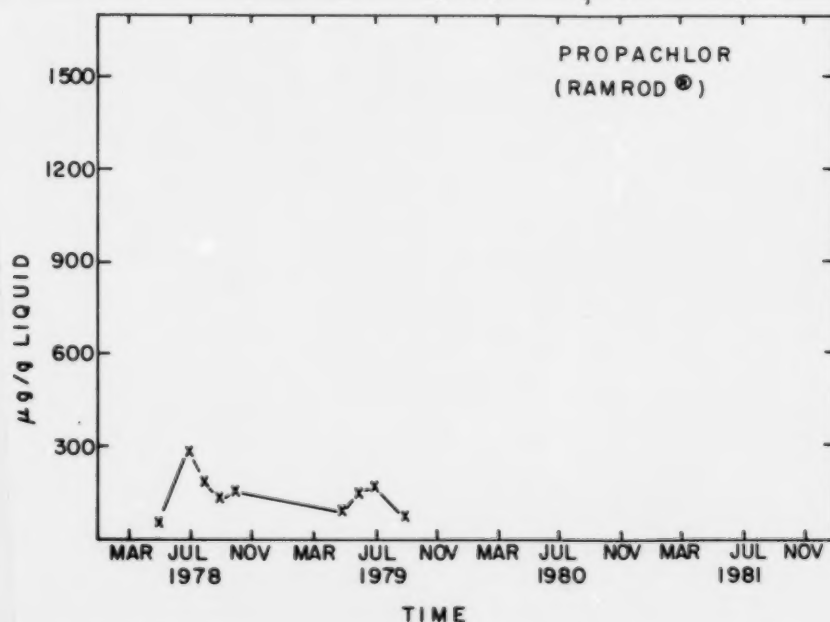


Figure 66. Propachlor concentration in liquid samples from agronomy macropit #2.

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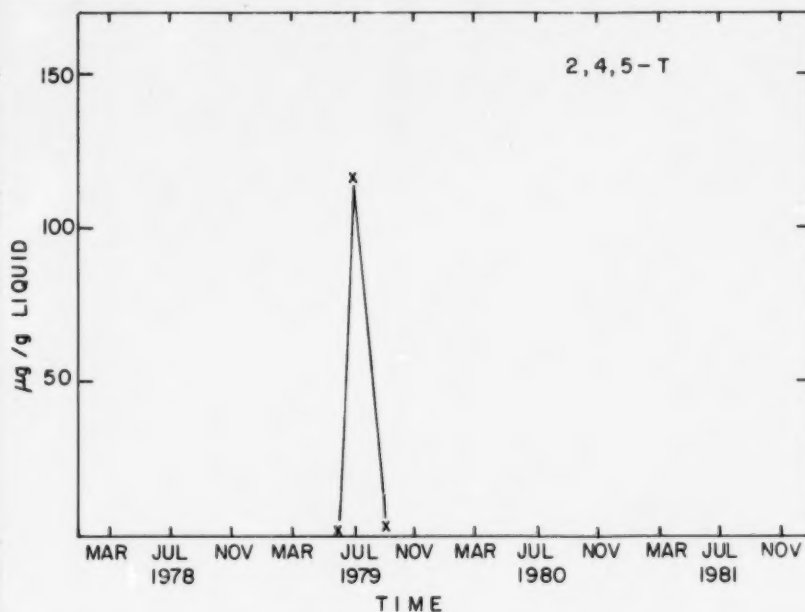


Figure 67. 2,4,5-T concentration in liquid samples from agronomy macropit #2.

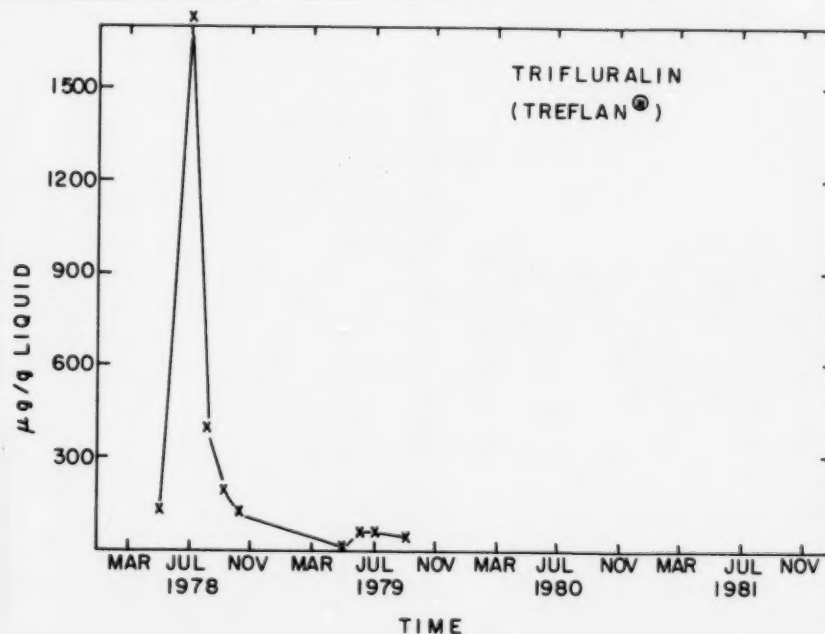


Figure 68. Trifluralin concentration in liquid samples from agronomy macropit #2.

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Figures 49-68 where the amounts measured on each sampling date are plotted separately for each pesticide which is ordered alphabetically for ease of location. The formulations are shown in parentheses. Originally, these graphs were produced with the intention of conducting a monitor program over a period of four years to end in the fall of 1981. This explains the extended horizontal axes. The vertical axes are either 0-15, 0-150 or 0-1500 $\mu\text{g/g}$ of liquid sample depending on the maximum concentration observed for any one residue in the liquid phase. In some cases as with Basagran in Figure 52, the data points begin sometime after the initiation of the sample and monitor program in May 1978 because analytical procedures had not yet been developed for the measurement of the listed pesticide.

As with the Horticulture macropit data, the connection of these data points with straight lines is not a valid representation of decay because of the uncontrolled manner in which the pesticides are deposited. The plots do however give a quick visual representation of possible build-up of any pesticide. The reader is referred to the Horticulture Macropit section for a discussion of why these data are not massaged by the deposition data in Table 22.

In some instances, as with 2,4-D and 2,4,5-T, measurable amounts of pesticides are present yet no records of deposition are available. These observations focus sharply on the problem of providing completely accurate disposal data for any disposal system which is economically practical.

Surroundings - Agronomy Farm

Water and air samples were taken in the vicinity of the disposal pits located on the Agronomy farm and at sites remote from these pit systems. The approach was much the same as that described for the Horticulture farm. These water and air analyses data are reported and discussed separately in the following sections. No appreciable contamination of either the surrounding water or the air can be attributed to the presence of the disposal pits.

Water and Soil Analysis--

The results of the analyses of ground water samples taken at the Agronomy farm are given in Table 24. No tile surrounds this plastic lined pit so the May 31, June 15 and June 28 samples were taken from the ground water seepage into 10 cm holes augered at each of the four outside corners of the pit. These data suggest some seepage of chemicals into the ground water immediately adjacent to the pit but the long range effect of this probable leakage is uncertain. Additional data will also be necessary to confirm positively the extent of the chemical losses by the proposed route.

The Agronomy macropit #1 had been in use for thirteen years so some shallow ground samples at a depth of ~ 3 meters were taken at sites located about midway between the two macropits which are separated by only about 50 yards. At this location one should be able to measure contamination from either pit system. Some pesticide residues were measured but the amounts are similar to those observed from water samples taken from the field located

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TABLE 24. AMOUNTS OF HERBICIDES IN PPM IN GROUND WATER
OUTSIDE THE AGRONOMY MACRO PIT #2

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Herbicide	May 31 1979	Jun 15 1979	Jun 28 1979	Jun 5 1979 Between Pits	Jun 5 1979 Near-by Field
EPTC (Eptam)	0.03	- *	0.01	-	-
Butylate (Sutan)	0.11	-	0.04	-	-
Chlorpropham (CIPC)	-	-	<0.01	-	-
Propachlor (Ramrod)	-	-	-	-	-
Atrazine (Aatrex)	-	-	0.28	-	-
Trifluralin (Treflan)	-	-	-	0.05	<0.01
Dimethoate (Cygon)	-	-	-	-	-
Alachlor (Lasso)	-	-	0.03	0.05	<0.01
Diphenamid (Dymid)	-	-	-	-	-
Cyanazine (Bladex)	-	-	<0.01	<0.01	<0.01
Metribuzin (Sencor)	0.22	0.31	0.34	<0.01	<0.01
Basagran [†]	2.5	2.8	15	<0.01	0.04
Dicamba [†]			2.1	<0.01	<0.01
2,4-D [†]	0.10	0.06	0.06	<0.01	<0.01
2,4,5-T [†]	0.25	0.03	8.8	<0.01	<0.01

* Not detected at <0.01 PPM

† Measured as methyl ester

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near-by. The interpretation of these two sets of results leads to the conclusion that neither pit is a source of contamination of ground water beyond that which occurs in agricultural areas due to normal pesticide applications and leachings.

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Soils removed during the augering operation at the corners of macropit #2 were composited and portions were analyzed for pesticide residues. No pesticides were found at the rather high detection limit of 10 µg/g soil. Further investigation of this unexpected phenomena of pesticides being present in the seepage water but not the surrounding soil is required. Apparently, the migration of pesticides capable of seeping from the disposal pit requires a cone of depression which can be very shallow. Deeper cones may lead to very much more significant migration through possible ruptures and other leakage paths in the plastic liner.

Air Analyses--

The results of the analysis of the air taken above and upwind from macropit #2 are given in Table 25, 26 and 27 for June 28, August 14 and August 24, 1979, respectively. The interpretation of these results relative to containment are the same as that discussed for the Horticulture macropit.

The evidence for pesticide losses due to evaporation is as expected for EPTC, butylate, trifluralin and alachlor. All the other evidence of losses due to evaporation from the pit surface is very questionable due to similar results for analyses of samples taken upwind and above the pit.

Agronomy Minipits

Investigators from the departments of Entomology, Bacteriology, and Agricultural Engineering and the Energy and Mineral Resources Research Institute (EMRRI) formulated a plan in late 1976 for the development of guidelines for the safe disposal of pesticides in minipit systems suitable for use by individual farmers. Simplicity, low cost and infrequent maintenance of workable systems were the major guidelines used in the experimental design. The interpretation of results from these studies was also intended to be applicable to larger macropit systems.

A field experiment was designed to determine if small amounts of selected pesticides would degrade in inexpensive disposal pits containing soil and water at ambient temperatures. The containers for the minipit studies were 110 liter plastic garbage cans buried partially in the ground and filled with 60 liters of water and 15 kilograms of soil.

Six formulated pesticides, atrazine, (Aatrex®; 80W), alachlor (Lasso®; E.C.), 2,4-D ester (Weedone®; LV4; E.C.), trifluralin (Treflan®; E.C.), carbaryl (Sevin®; 50W), and parathion (Security®; 15W), were studied individually and as a mixture at the two concentrations of 0.5 and 0.025% active ingredient by weight based on 60 liters of water; i.e. 300 and 15 g, respectively. Additional variables of aeration at one liter per minute and peptone nutrients at 0.1% by weight resulted in a factorial experiment of 56 systems.

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TABLE 25. AMOUNTS OF HERBICIDES IN ng/l AIR SAMPLES COLLECTED
AT THE AGRONOMY DISPOSAL PIT JUNE 28, 1979

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Herbicide	Up-Wind from Pit	On Top† of Pit
EPTC	*	0.6
Butylate	*	0.3
Chlorpropham	<0.1	*
Propachlor	*	<0.1
Atrazine	<0.1	<0.1
Trifluralin	<0.1	0.3
Dimethoate	*	*
Alachlor	0.2	1.5
Diphenamid	*	*
Cyanazine	<0.1	<0.1
Metribuzin	<0.1	0.1

26
TABLE 26. AMOUNTS OF HERBICIDES IN ng/l AIR SAMPLES COLLECTED
AT THE AGRONOMY DISPOSAL PIT AUG. 14, 1979

Herbicide	Up-Wind from Pit	On Top† of Pit
EPTC	<0.1	1.0
Butylate	<0.1	<0.1
Chlorpropham	*	*
Propachlor	*	<0.1
Atrazine	*	*
Trifluralin	*	0.6
Dimethoate	*	*
Alachlor	*	0.5
Diphenamid	*	*
Cyanazine	*	*
Metribuzin	*	0.1

* Not detected

† Downwind corner of pit

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TABLE 27. AMOUNTS OF HERBICIDES IN ng/l AIR SAMPLES COLLECTED
AT THE AGRONOMY DISPOSAL PIT AUG. 24, 1979

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Herbicide	Up-Wind from Pit	On Top† of Pit
EPTC	<0.1	4.5
Butylate	*	1.2
Chlorpropham	*	*
Propachlor	*	*
Atrazine	*	*
Trifluralin	*	0.8
Dimethoate	*	*
Alachlor	*	0.9
Diphenamid	*	*
Cyanazine	*	*
Metribuzin	*	0.2

* Not detected

† Downwind corner of pit

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The formulated pesticides were added on April 27, 1977 to the minipits located within a fenced area at Agronomy and Agricultural Engineering Research Center (AAERC), referred to simply as the Agronomy farm elsewhere in this report.

Soil and Liquid Analyses--

Representative aliquots of the contents of all of the minipits were taken for analyses at intervals of 0, 1, 3, 4, 8, 12, 16, 20, 24, 28, 52, and 68 weeks after addition. The samples were analyzed for decay of the added pesticides and two hydrolysis products, 2,4-D acid and 1-naphthol.

The numerical data from these analyses were used to formulate decay graphs which are presented in one form in Figures 69-84 and in a second orientation in Figures 85-100. For the sake of clarity in the figures the analytical data from 1, 3 and 4 weeks have been averaged and plotted as a single result at the four week interval. These figures indicate vividly which pesticides degrade and what factors such as concentration, aeration, mixtures, and nutrients affect the rate of degradation. The graphs also indicate the inevitable uncertainty in the analytical results due to sample heterogeneity.

The placement of the decay graphs in Figures 69-84 show best the effect of concentration, where several pesticides decay readily at low concentration but do not show measurable decay when present at high concentration. This placement of decay graphs also combines all the data and conditions for decay of a single pesticide on two figures. The first figure contains eight individual graphs and shows the results when only one pesticide is present. The second has an equal number of graphs showing the decay of the same pesticide in the presence of five others added at the same concentration of active ingredient. These figures are useful for highlighting the most favorable conditions for decay of an individual pesticide but they do not provide a good visual representation of the decay of the pesticides relative to each other. This is best accomplished by referring to the orientation of Figures 85-100.

Figures 85-92 show the decay of the six pesticides and the two hydrolysis products under the different test conditions when small amounts (15 g) are being disposed of. This representation indicates clearly which pesticides decay and which are persistent. Each figure represents a different condition with all permutations being considered. The same representations for larger amounts (300 g) are given in Figures 93-100.

The individual decay graphs in Figures 69-84 can be used to obtain detailed information about the decay of the various pesticides at any one point in time. These comparisons, when weighed properly against the analytical uncertainty, can be used to make deductions about the effects of the variables included as part of the minipit study. For example, the addition of peptone nutrient has a more pronounced effect on the rapid degradation of parathion (see Figure 79) than does aeration. However 15 g amounts of parathion are completely degraded within eight weeks at ambient conditions

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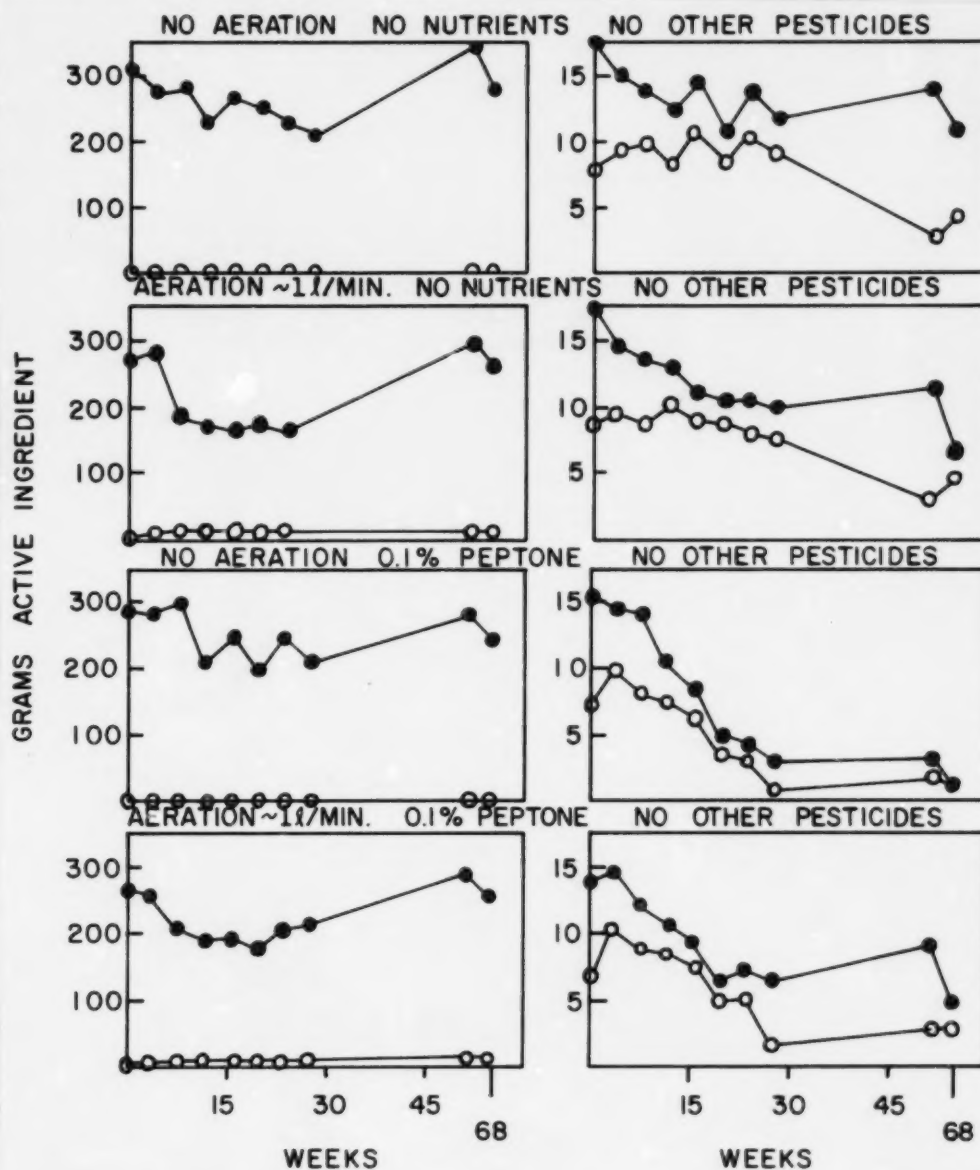


Figure 69. Alachlor decay from addition on 4/27/77 of 15 g or 300 g active ingredient as a Lasso[®] formulation per 60 l H₂O and 15 kg soil.

Aeration and nutrient variables listed above graphs.

● = Amount in water and soil. ○ = Amount in water.

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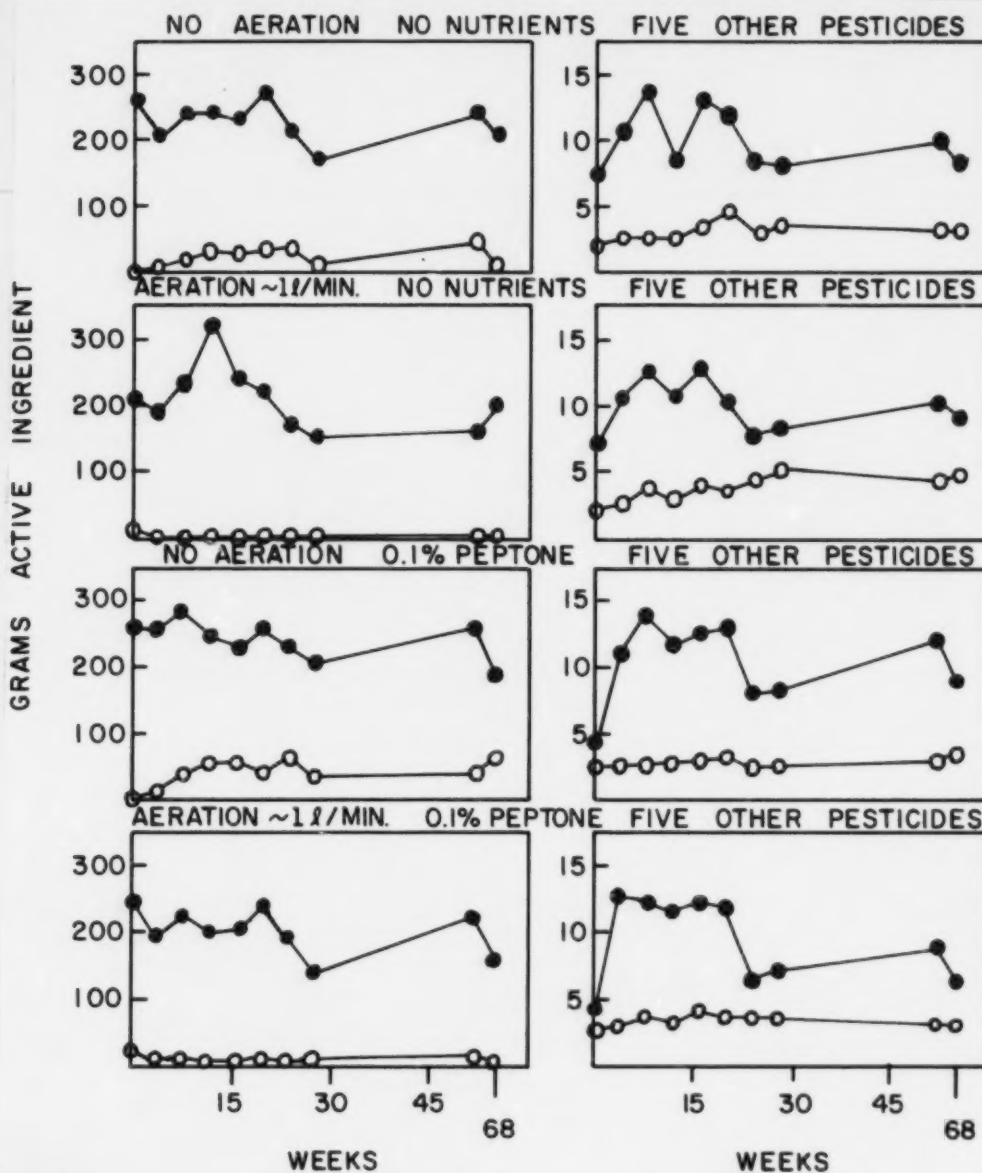


Figure 70. Alachlor decay in mixtures containing 15 g or 300 g active ingredient as a Lasso® formulation and similar amounts of each of five other formulated pesticides, Aatrex®, Weedone®, Treflan®, Security®, and Sevin® per 60 l H₂O and 15 kg soil. Pesticides were added on 4/27/77. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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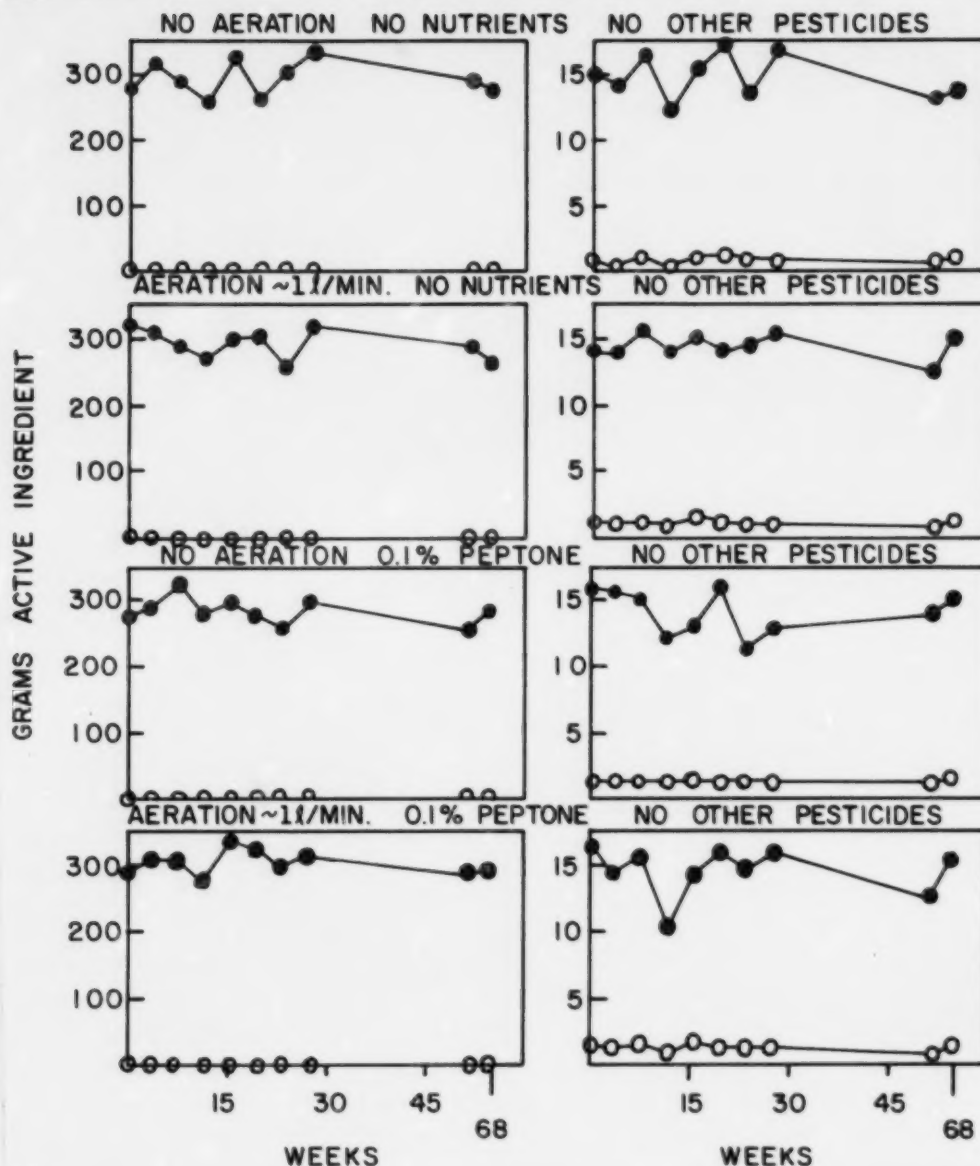


Figure 71. Atrazine decay from addition on 4/27/77 of 15 g or 300 g active ingredient as an Aatrex[®] formulation per 60 l H₂O and 15 kg soil.

Aeration and nutrient variables listed above graphs.
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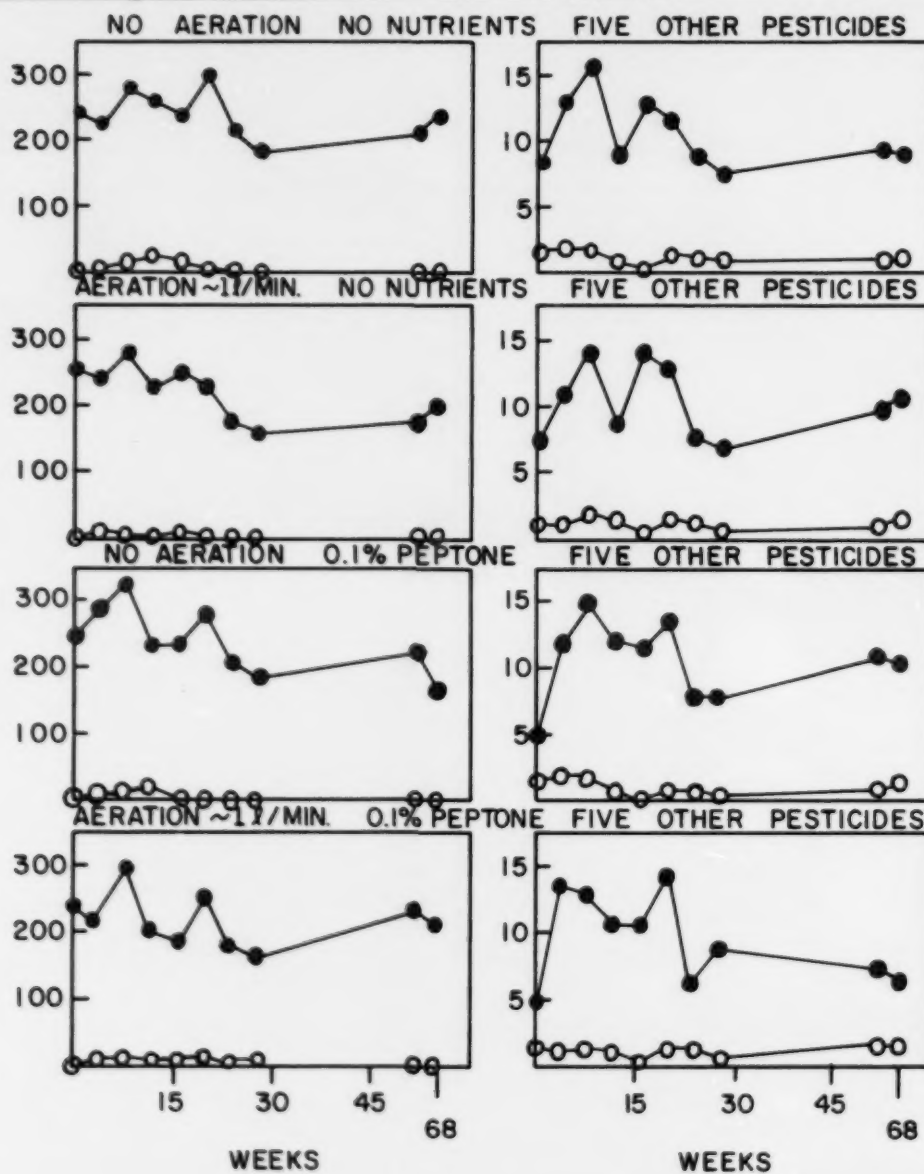


Figure 72. Atrazine decay in mixtures containing 15 g and 300 g active ingredient as an Aatrex® formulation and similar amounts of each of five other formulated pesticides, Lasso®, Weedone®, Treflan®, Security®, and Sevin® per 60 l H₂O and 15 kg soil.

Pesticides were added on 4/27/77.

Aeration and nutrient variables listed above graphs.

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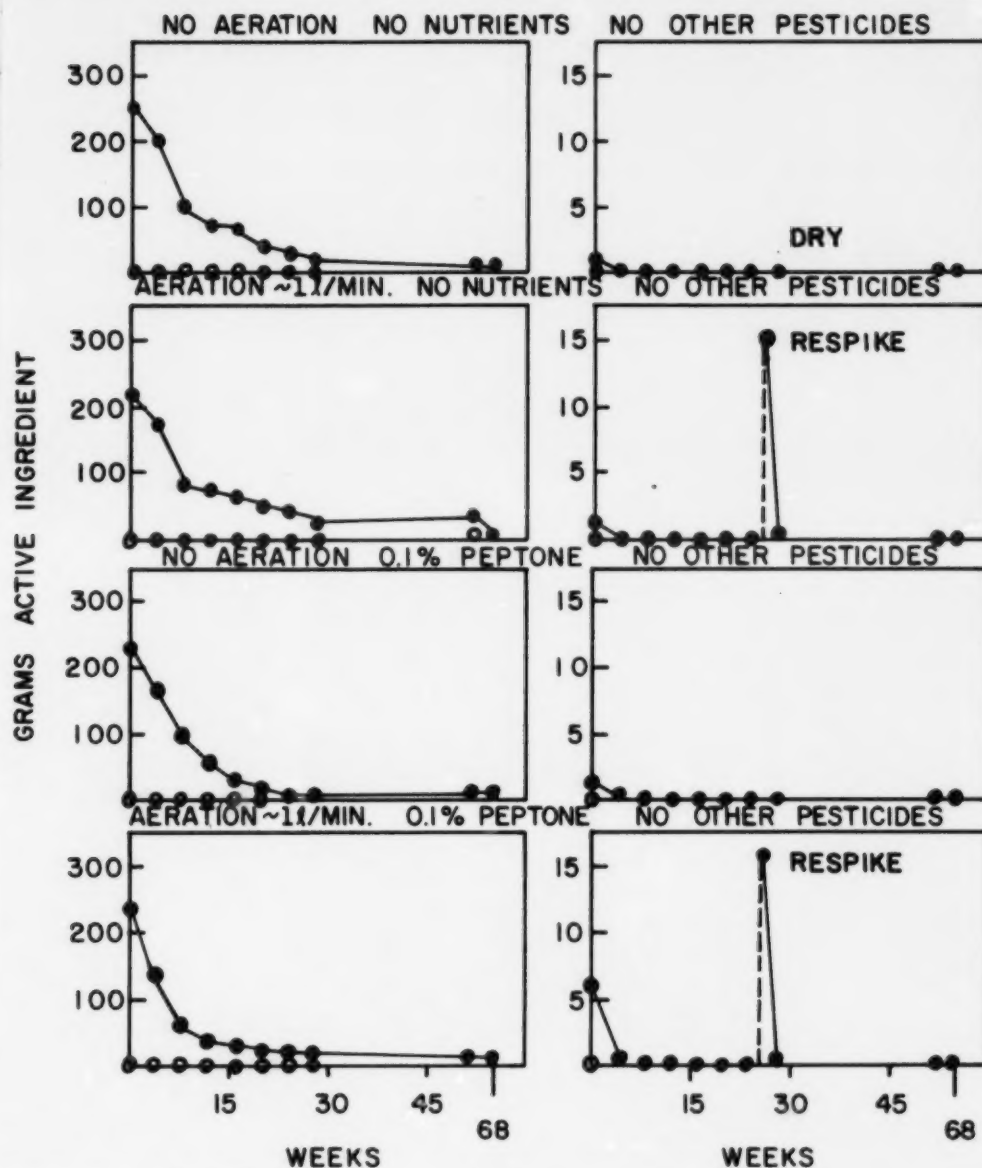


Figure 73. 2,4-D ester decay from addition on 4/27/77 of 15 g or 300 g active ingredient as a Weedone® formulation per 60 l H₂O and 15 kg soil.

Aeration and nutrient variables listed above graphs.

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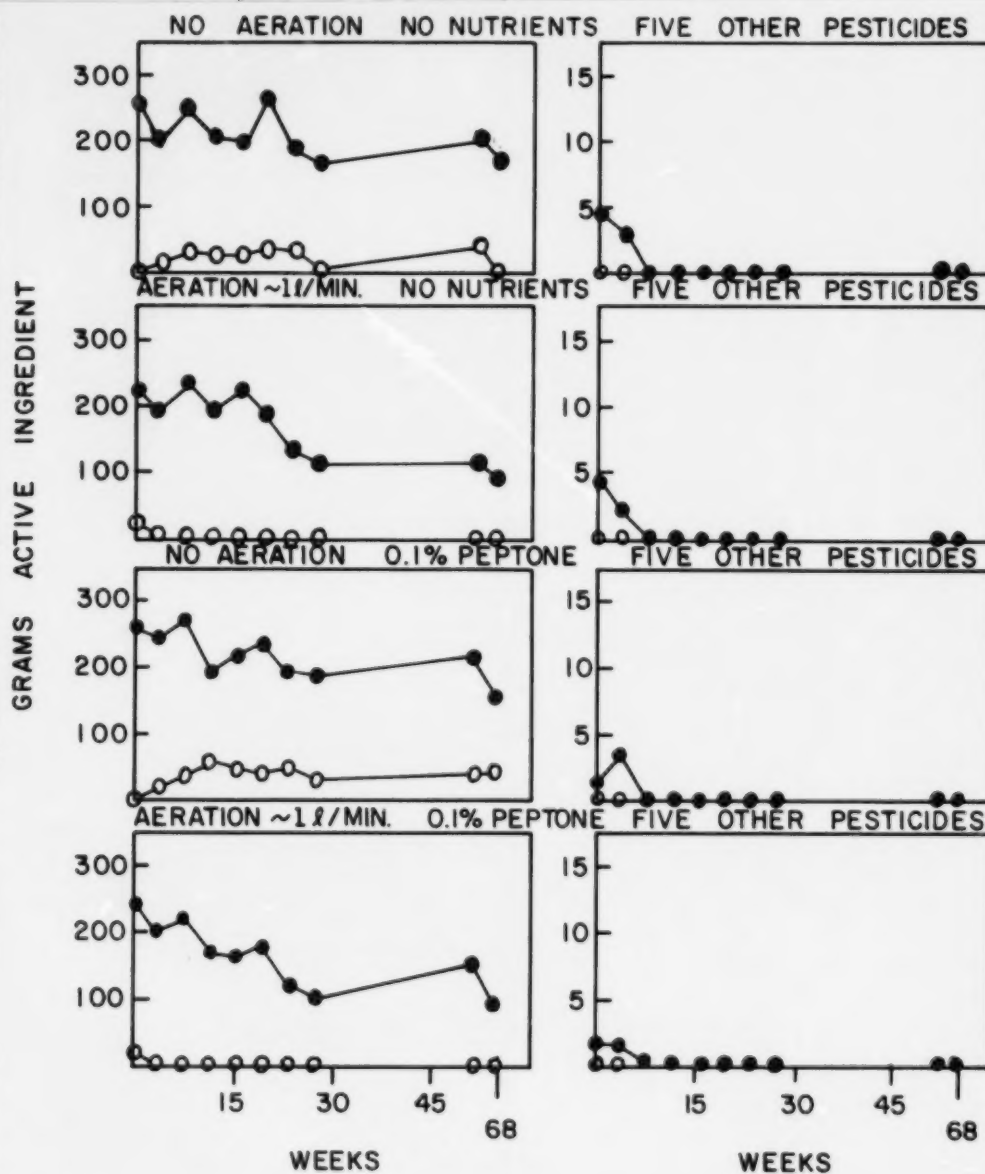


Figure 74. 2,4-D ester decay in mixtures containing 15 g or 300 g active ingredient as a Weedone® formulation and similar amounts of each of five other formulated pesticides, Lasso®, Aatrex®, Treflan®, Security®, and Sevin® per 60 l H₂O and 15 kg soil. Pesticides were added on 4/27/77. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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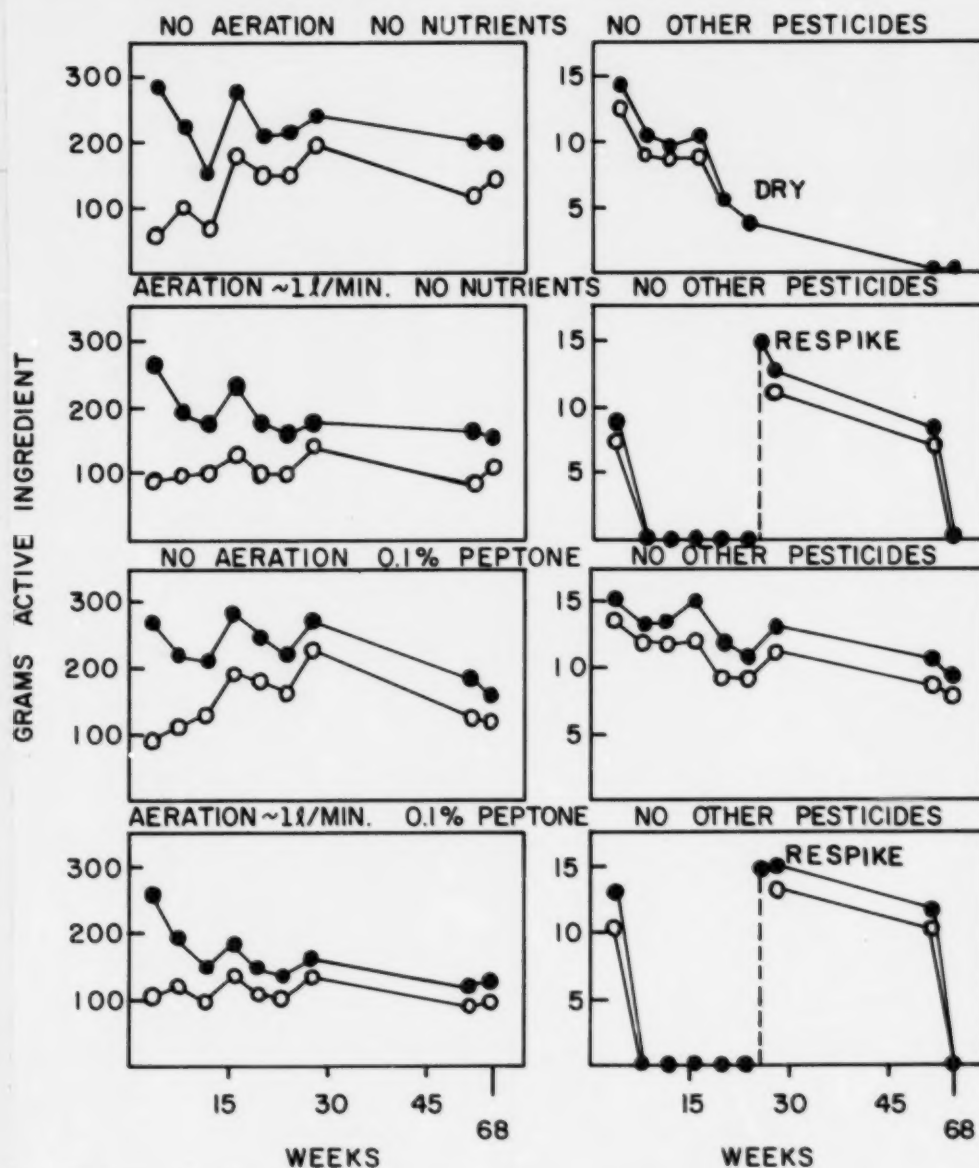


Figure 75. Plots of the decay of the 2,4-D ester plus the hydrolysis decomposition product, the 2,4-D acid. See the figure for the 2,4-D ester decay for the rest of this figure caption.

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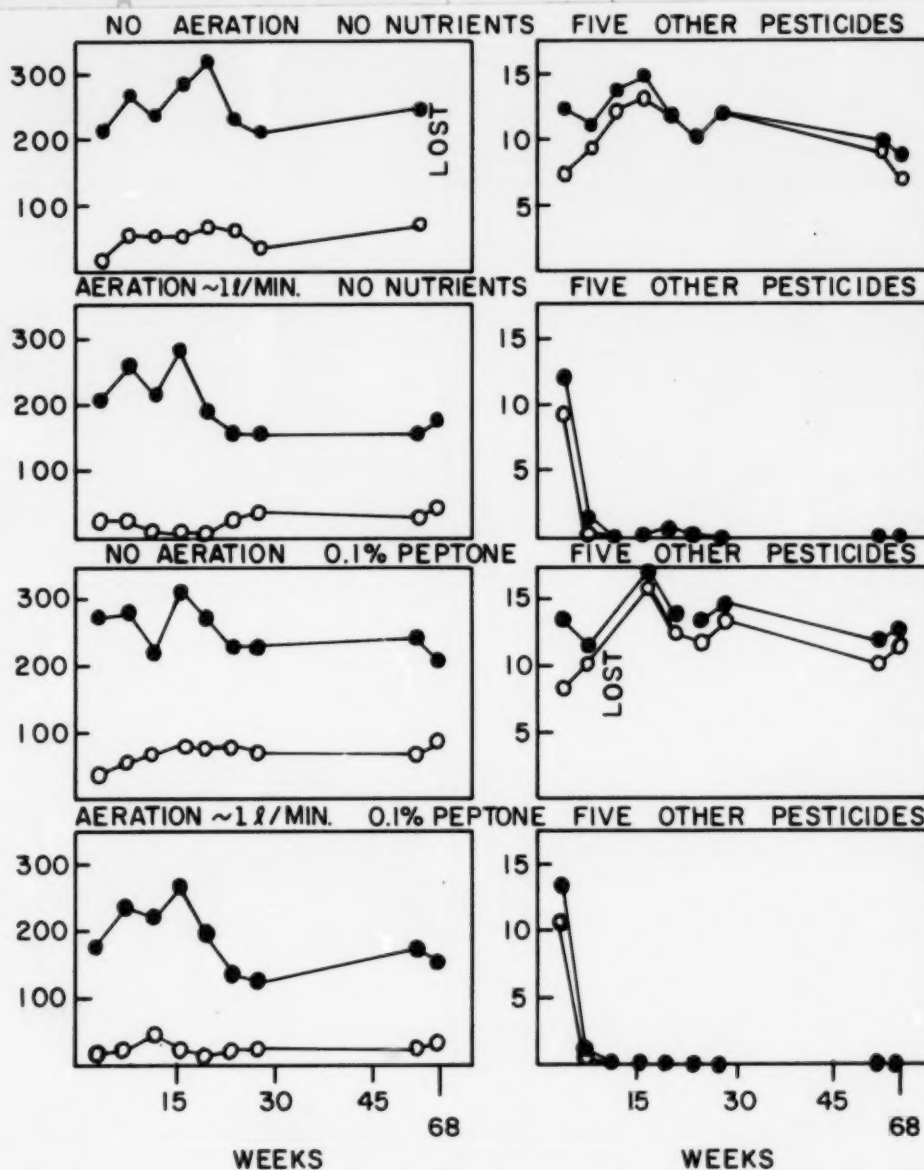
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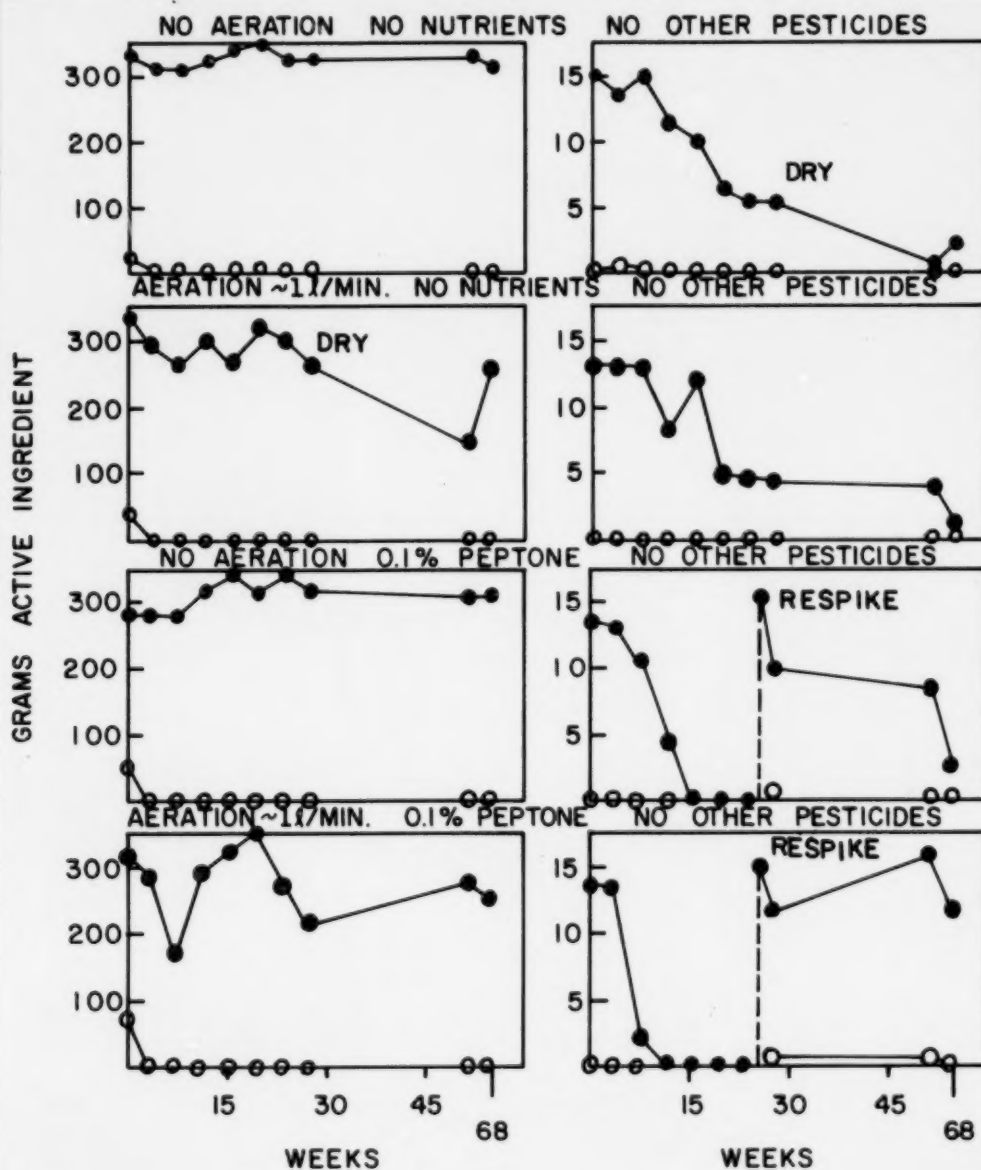


Figure 77. Trifluralin decay from addition on 4/27/77 of 15 g or 300 g active ingredient as a Treflan® formulation per 60 l H₂O and 15 kg soil. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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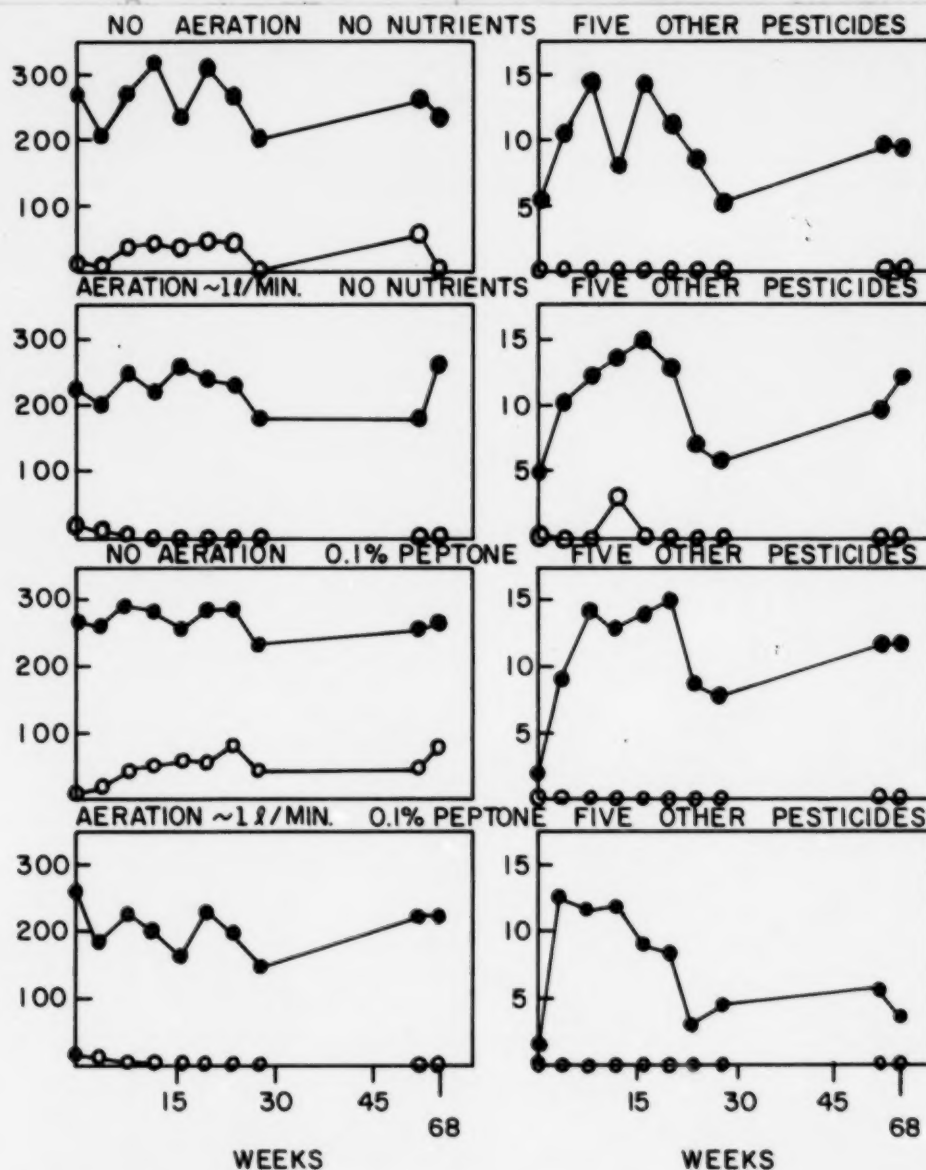


Figure 78. Trifluralin decay in mixtures containing 15 g or 300 g active ingredient as a Treflan® formulation and similar amounts of each of five other formulated pesticides, Lasso®, Aatrex®, Weedone®, Security®, and Sevin® per 60 l H₂O and 15 kg soil. Pesticides were added on 4/27/77. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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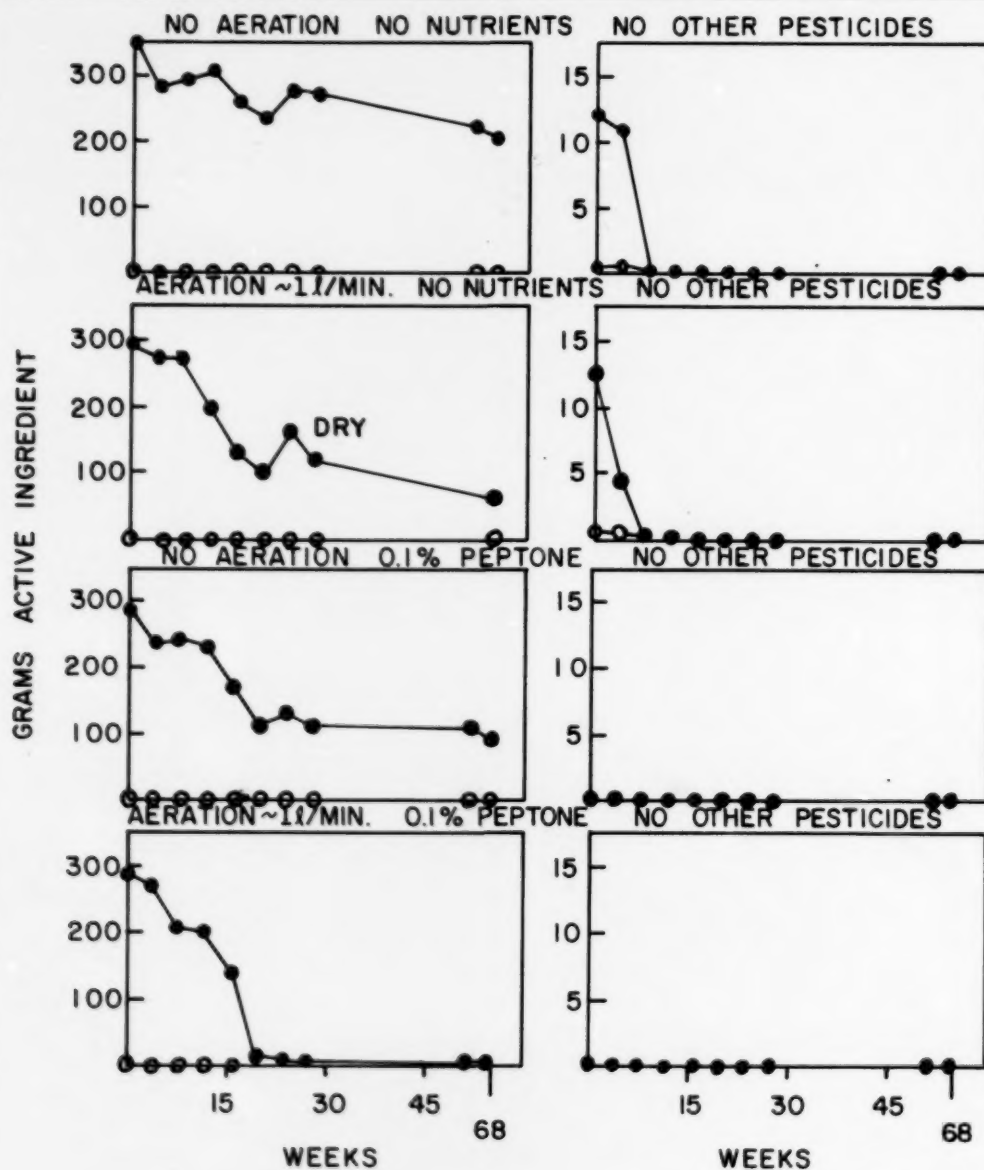


Figure 79. Parathion decay from addition on 4/27/77 of 15 g or 300 g active ingredient as a 15% Security® formulation per 60 l H₂O and 15 kg soil.

Aeration and nutrient variables listed above graphs.

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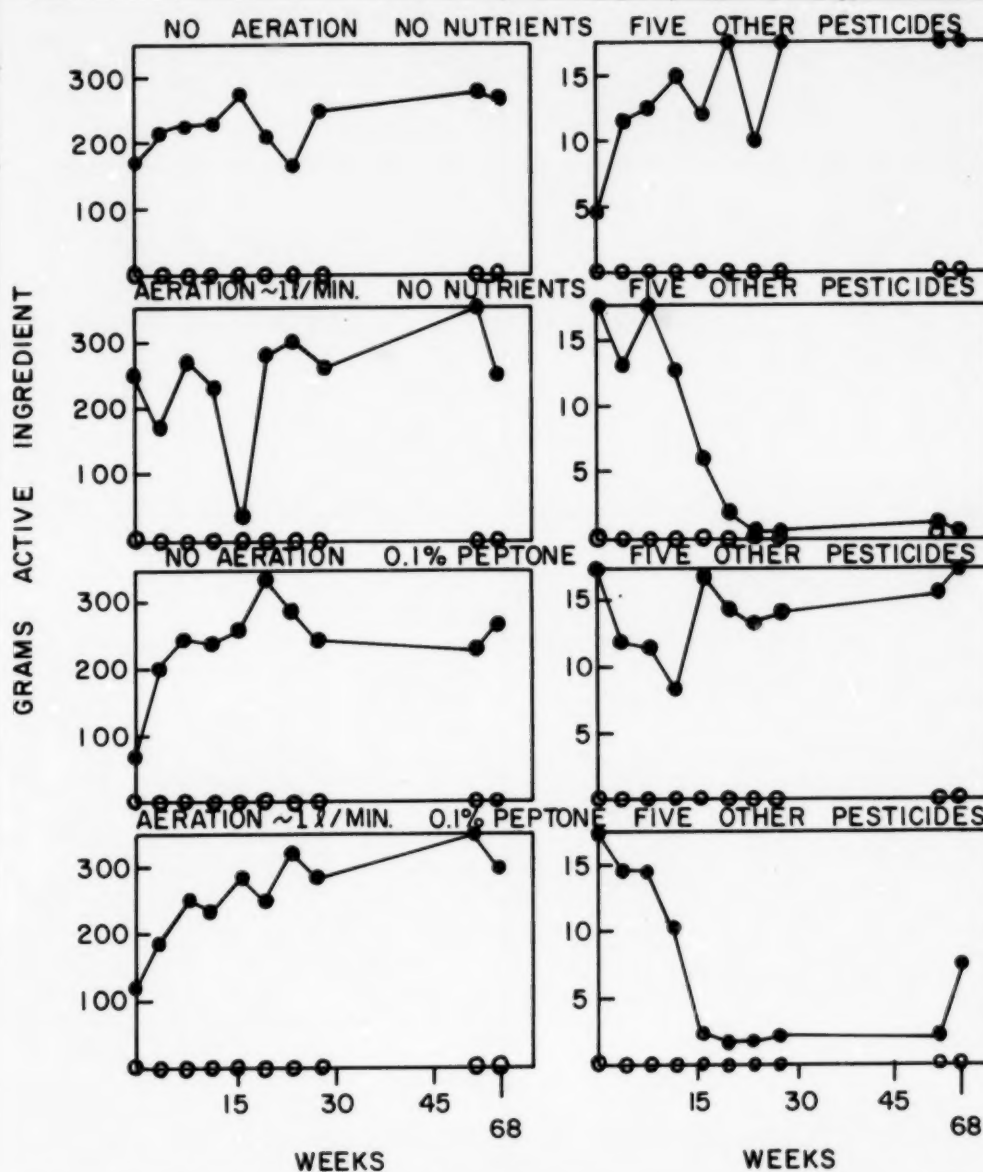


Figure 80. Parathion decay in mixtures containing 15 g or 300 g active ingredient as a Security[®] formulation and similar amounts of each of five other formulated pesticides, Lasso[®], Aatrex[®], Weedone[®], Treflan[®], and Sevin[®] per 60 l H₂O and 15 kg soil. Pesticides were added on 4/27/77. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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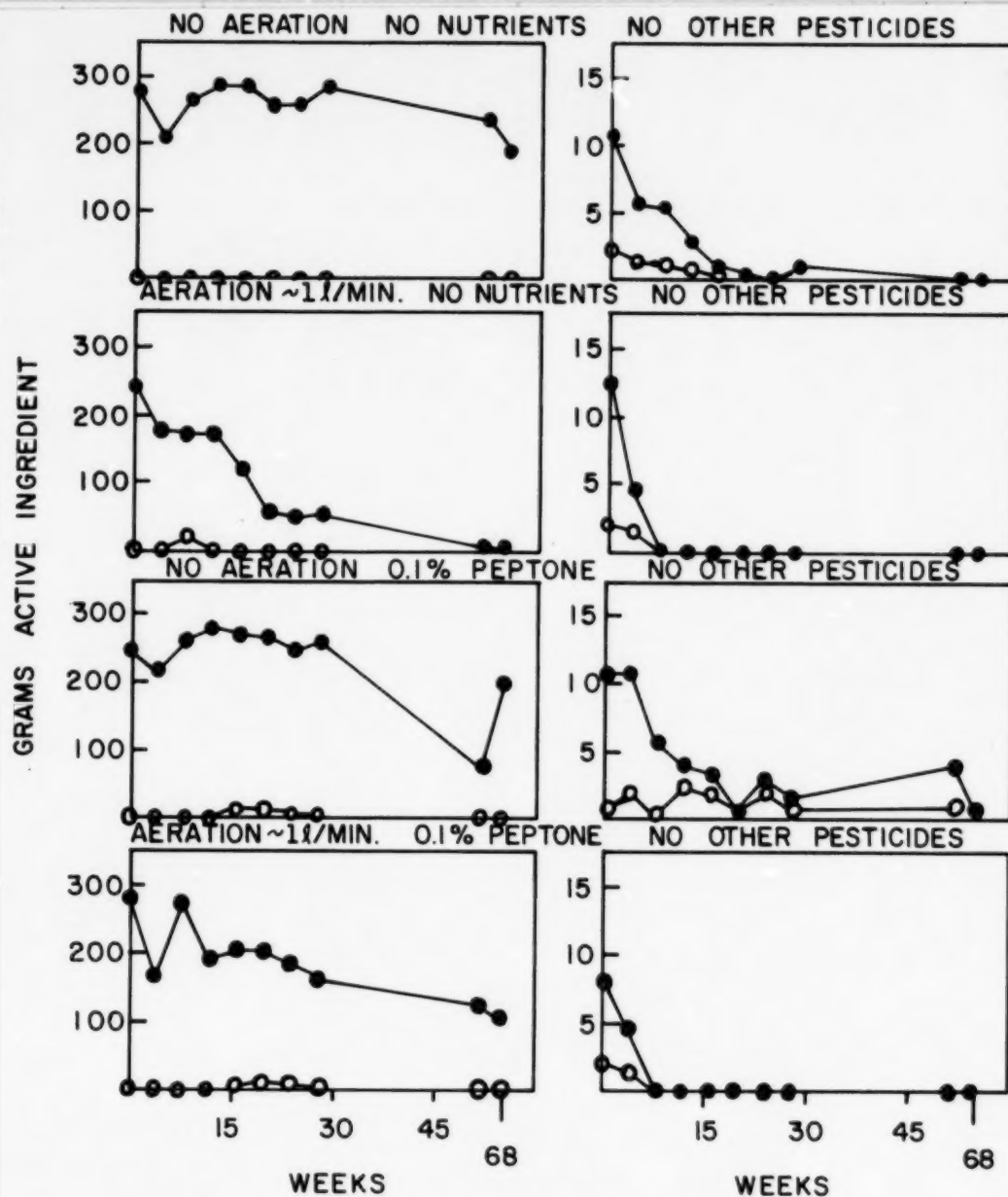


Figure 81. Carbaryl decay from addition on 4/27/77 of 15 g or 300 g active ingredient as a Sevin® formulation per 60 l H₂O and 15 kg soil. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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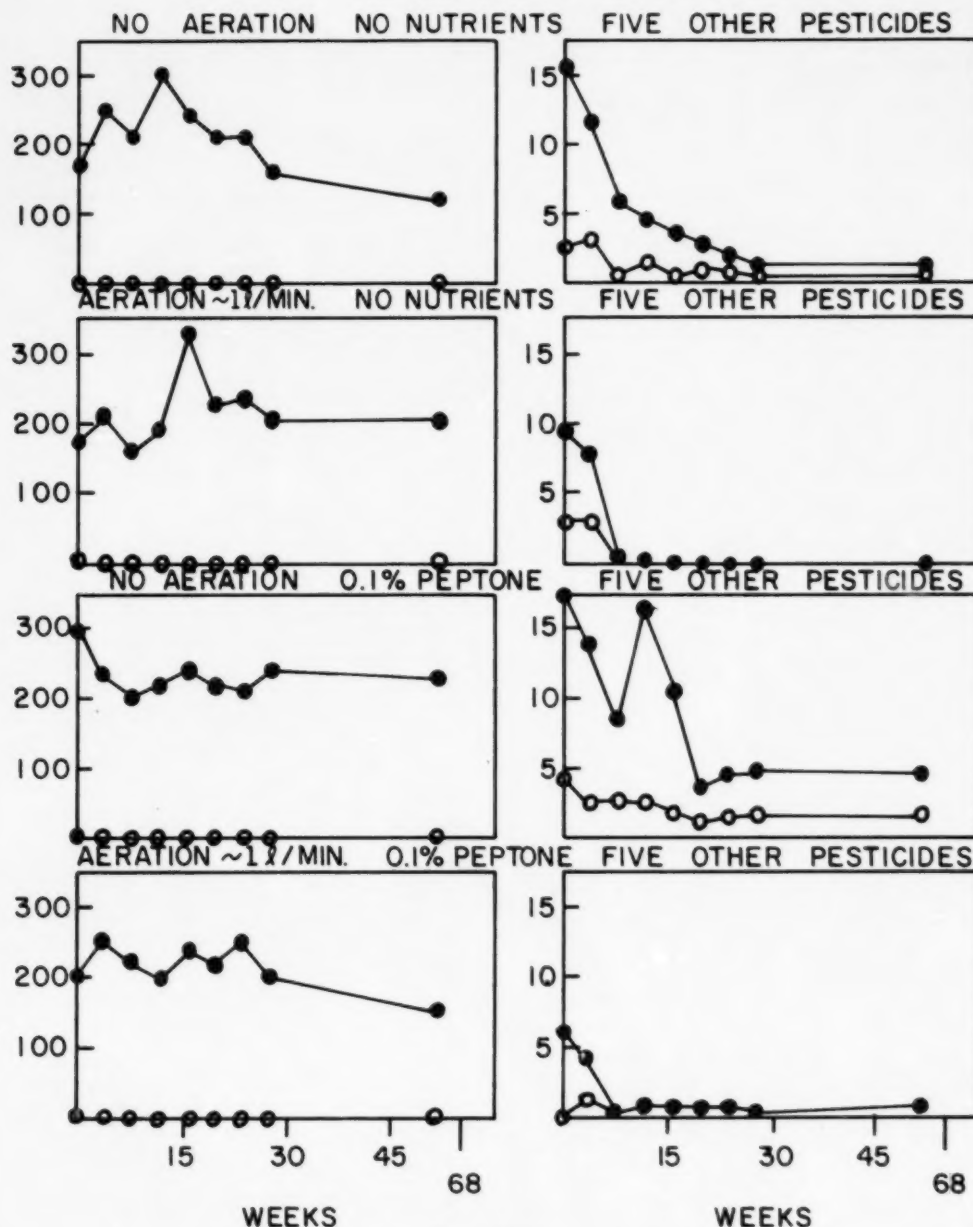


Figure 82. Carbaryl decay in mixtures containing 15 g or 300 g active ingredient as a Sevin® formulation and similar amounts of each of five other formulated pesticides, Lasso®, Aatrex®, Weedone®, Treflan®, and Security® per 60 l H₂O and 15 kg soil. Pesticides were added on 4/27/77. Aeration and nutrient variables listed above graphs. ● = Amount in water and soil. ○ = Amount in water.

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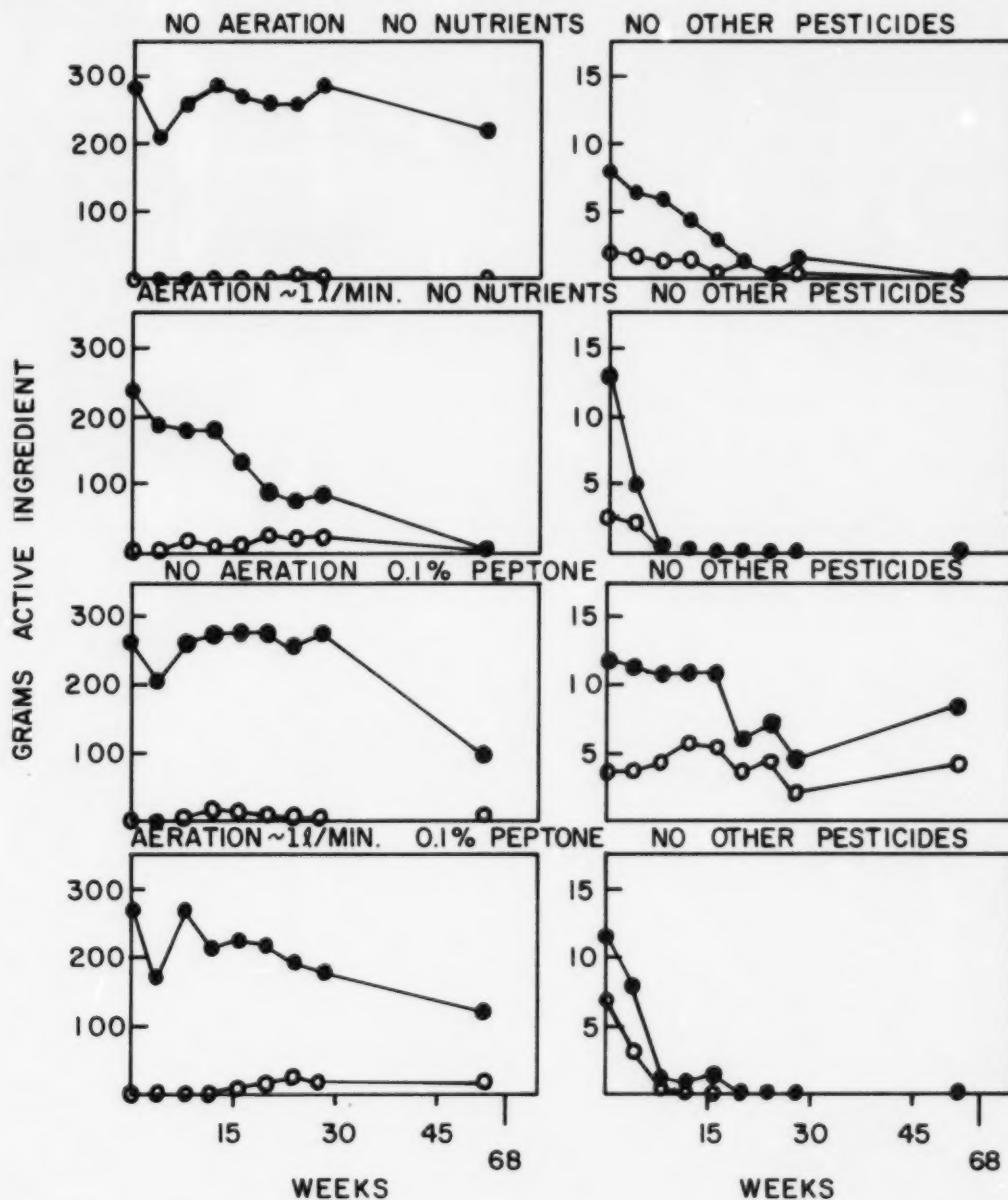


Figure 83. Plots of decay of carbaryl plus the decomposition product, 1-Naphthol. See the figure for the carbaryl decay for the rest of this figure caption.

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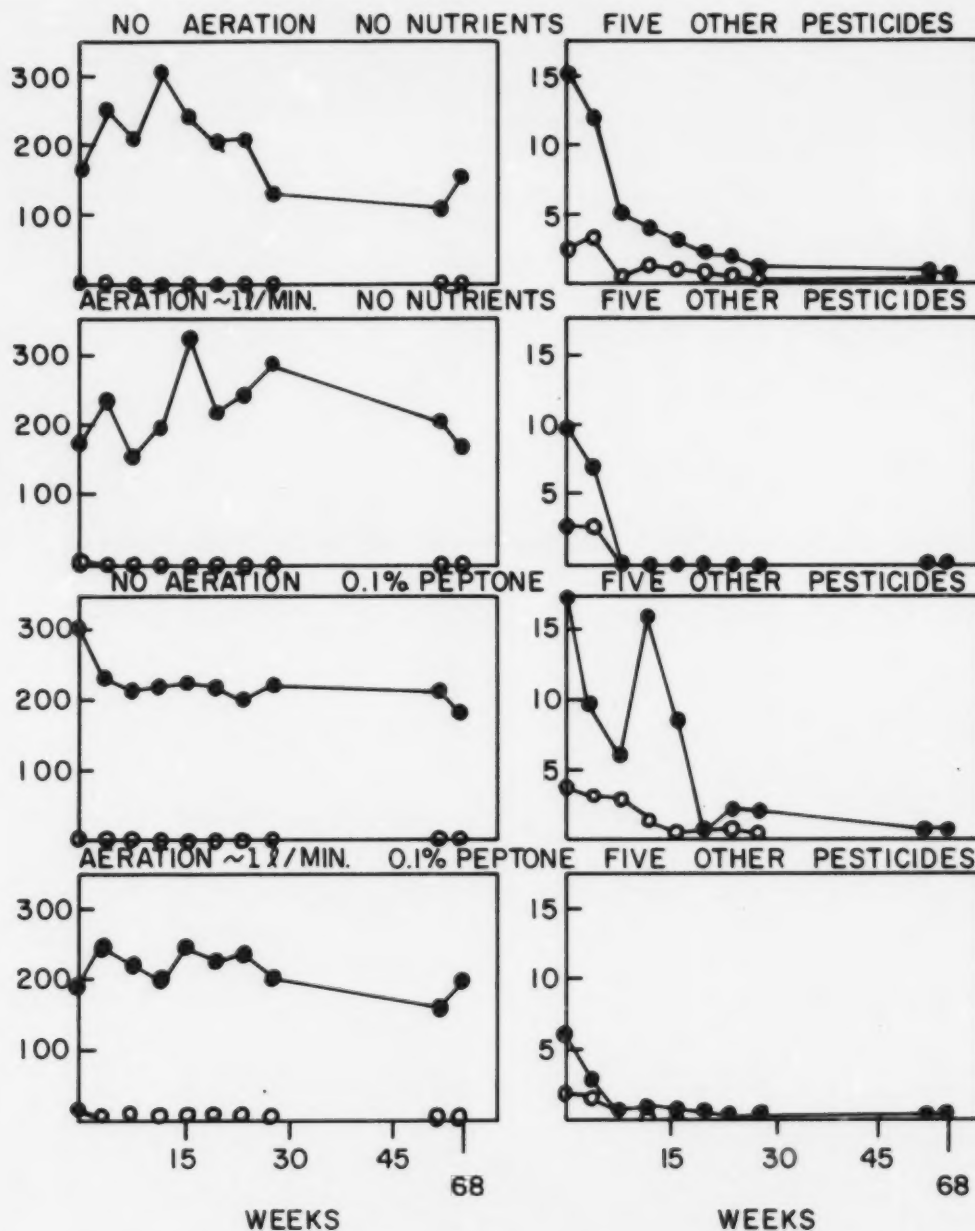


Figure 84. Plots of the decay of carbaryl plus the decomposition product, 1-Naphthol, in mixtures. See the figure for the carbaryl decay in mixtures for the rest of this figure caption.

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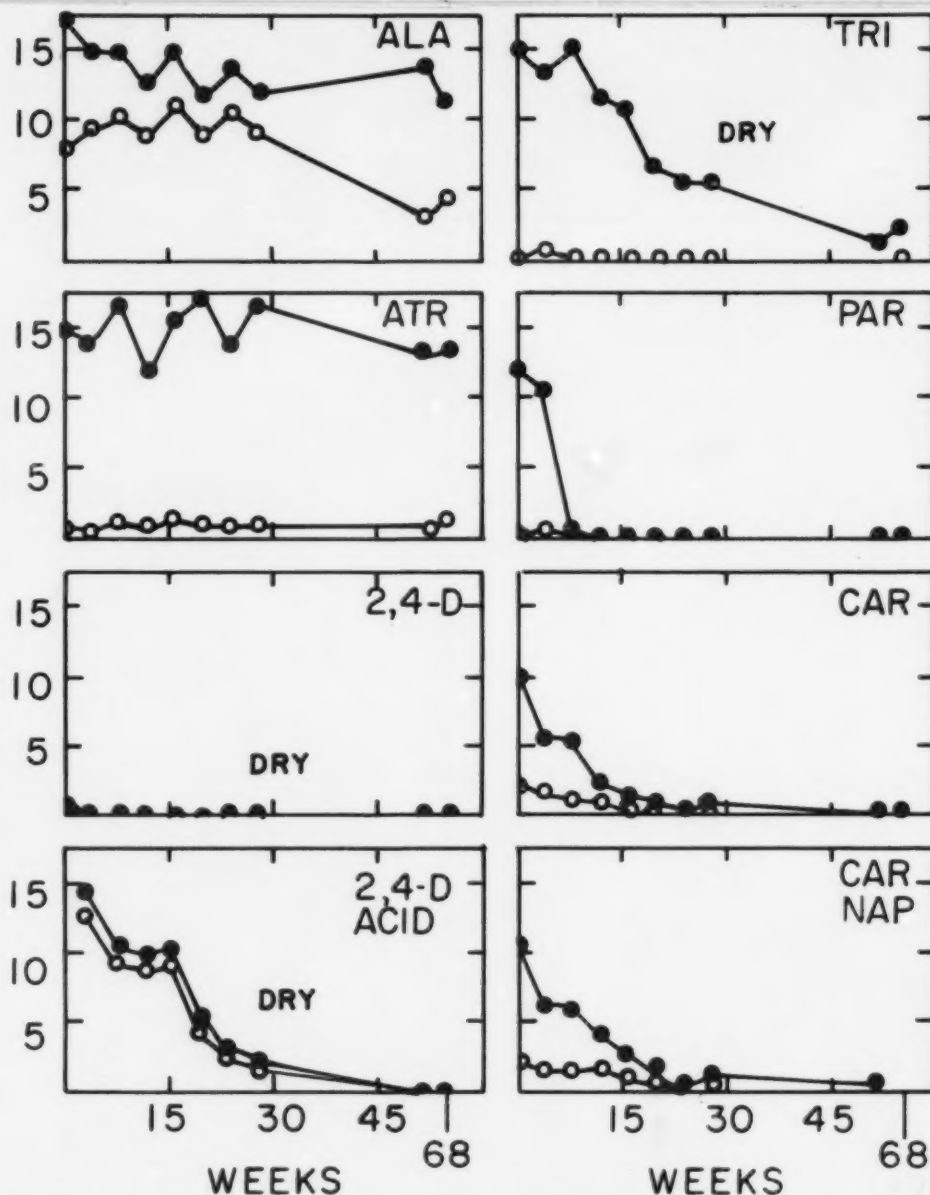


Figure 85. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).

● = Amount in water and soil. ○ = Amount in water.
No aeration, no nutrient, and no other pesticides.

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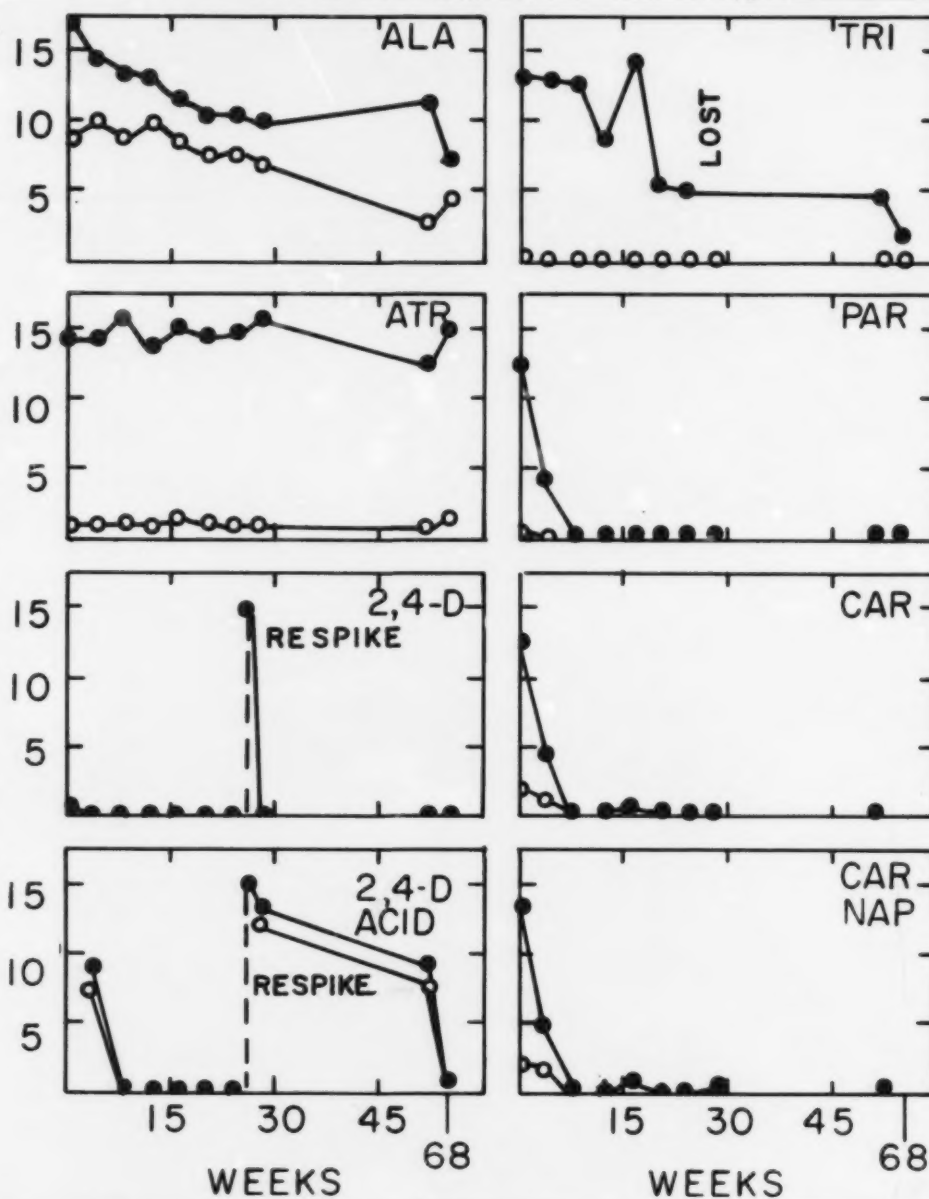


Figure 86. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parthion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
~ 1 l/min air, no nutrient, and no other pesticides.

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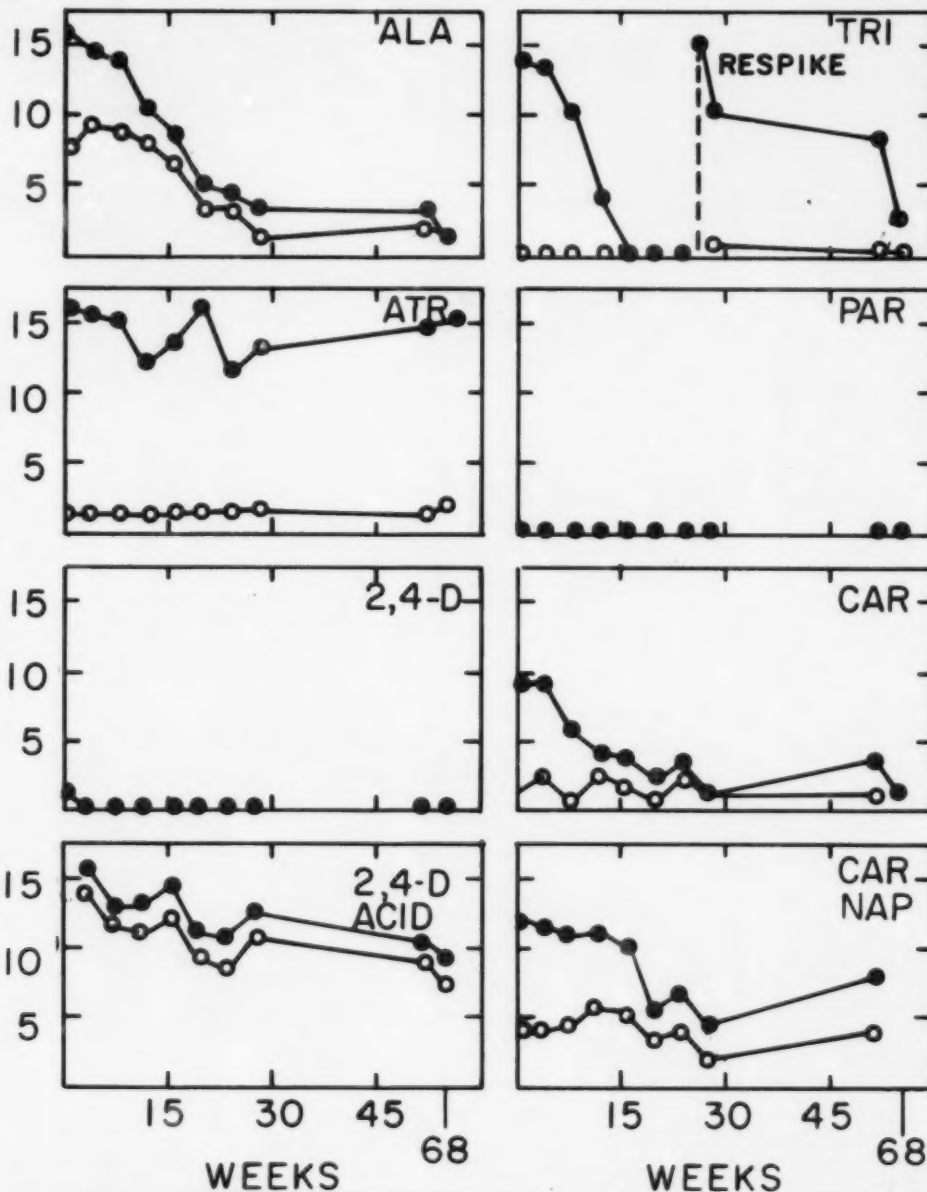


Figure 87. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
No aeration, 0.1% peptone nutrient, and no other pesticides.

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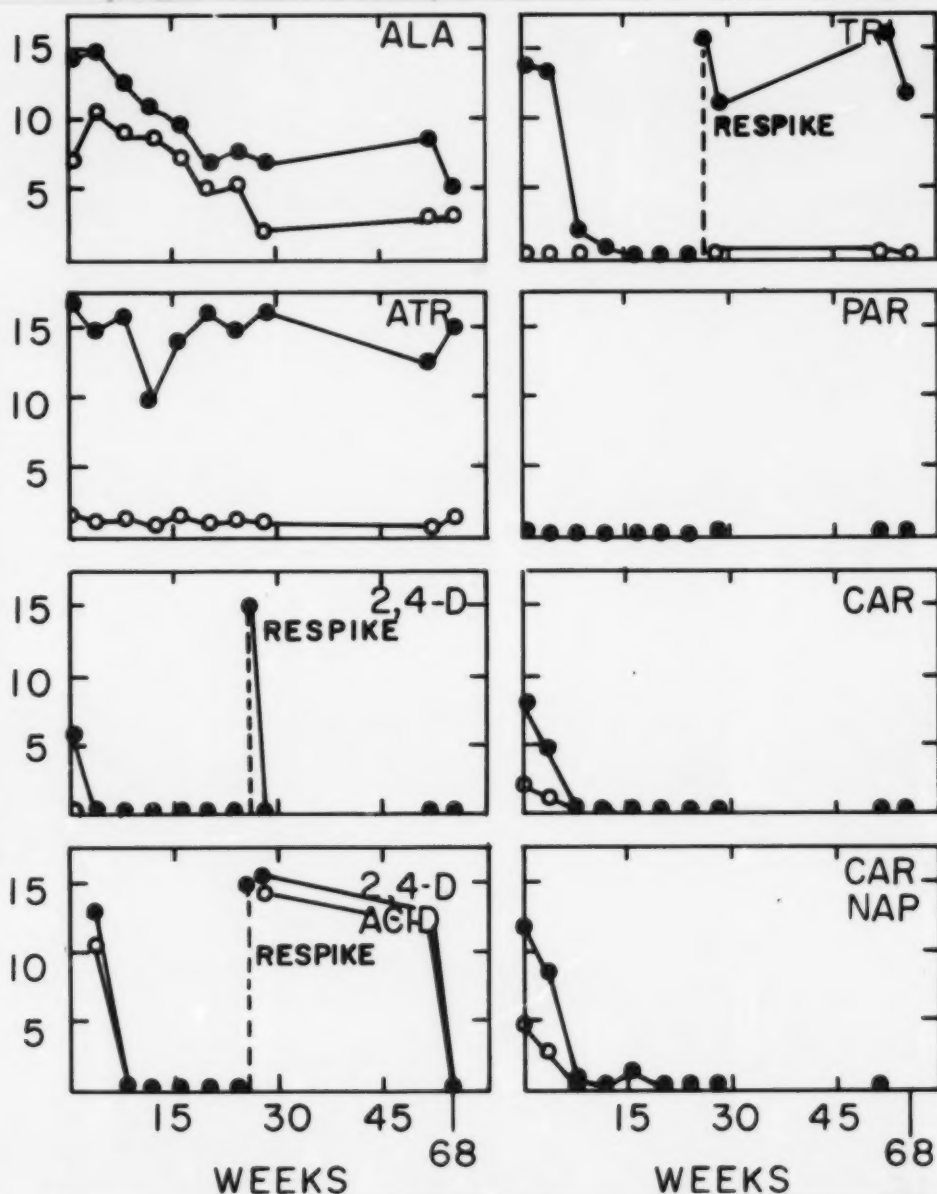


Figure 88. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol).
● = Amount in water and soil. ○ = Amount in water.
~1 l/min air, 0.1% peptone nutrient, and no other pesticides.

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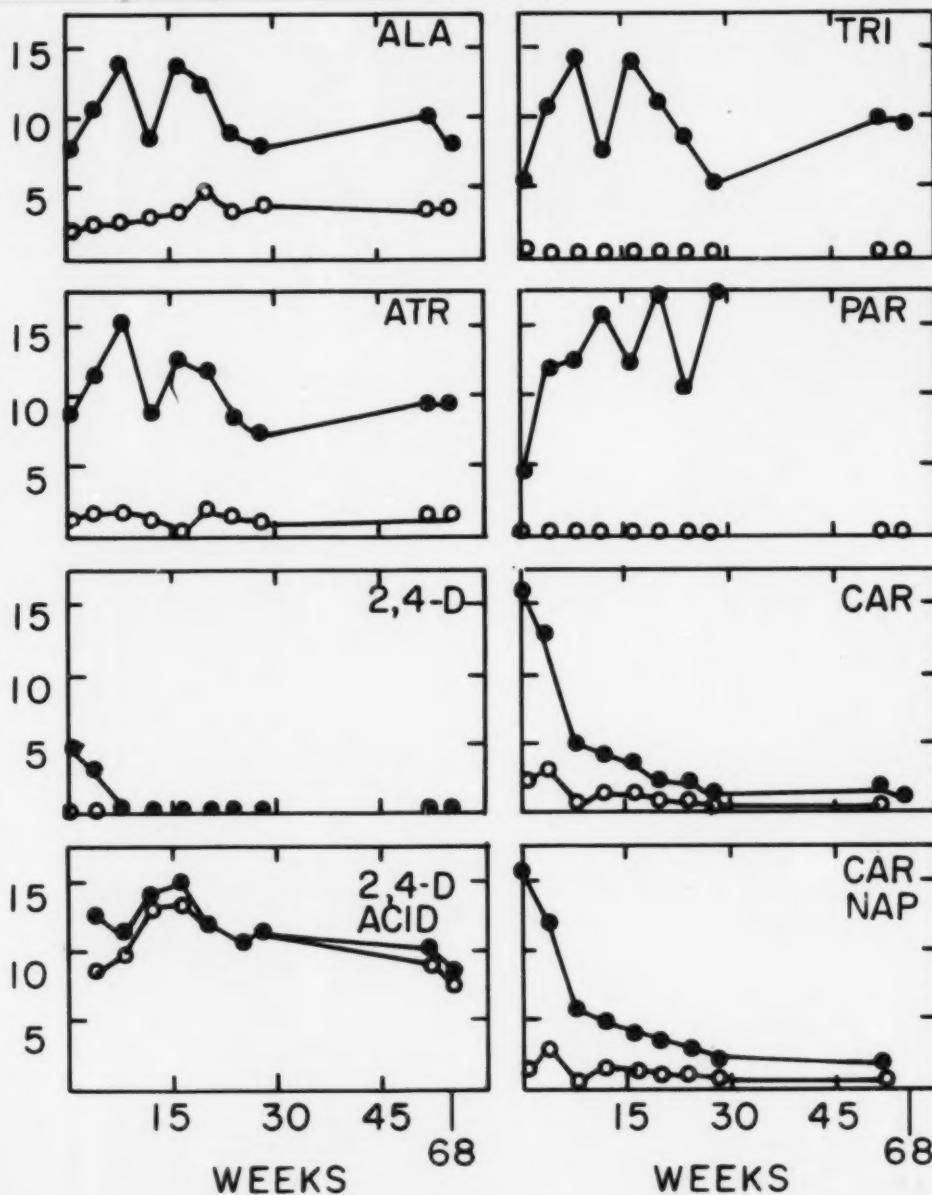


Figure 89. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, No nutrient, and 5 other pesticides.

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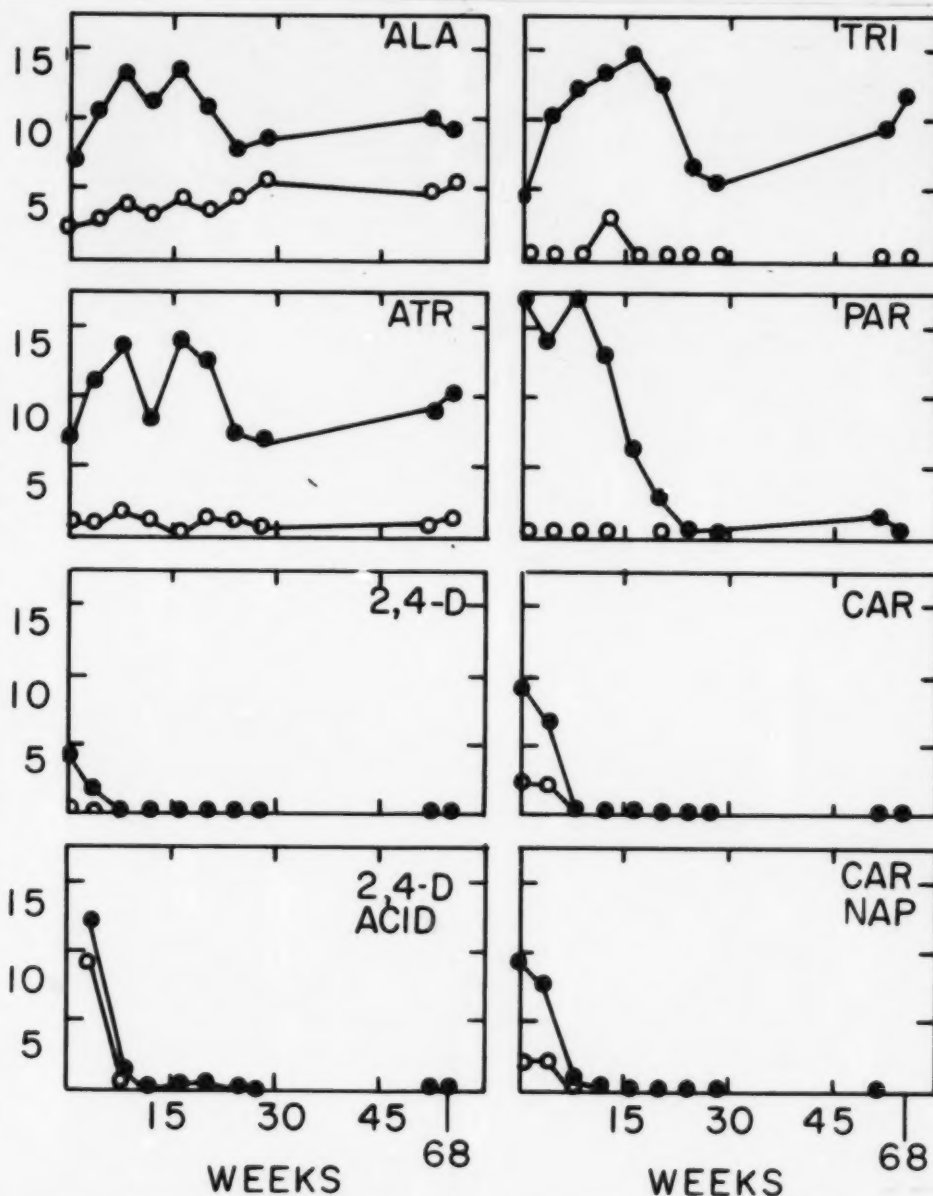


Figure 90. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min Air, No Nutrient, and 5 other pesticides.

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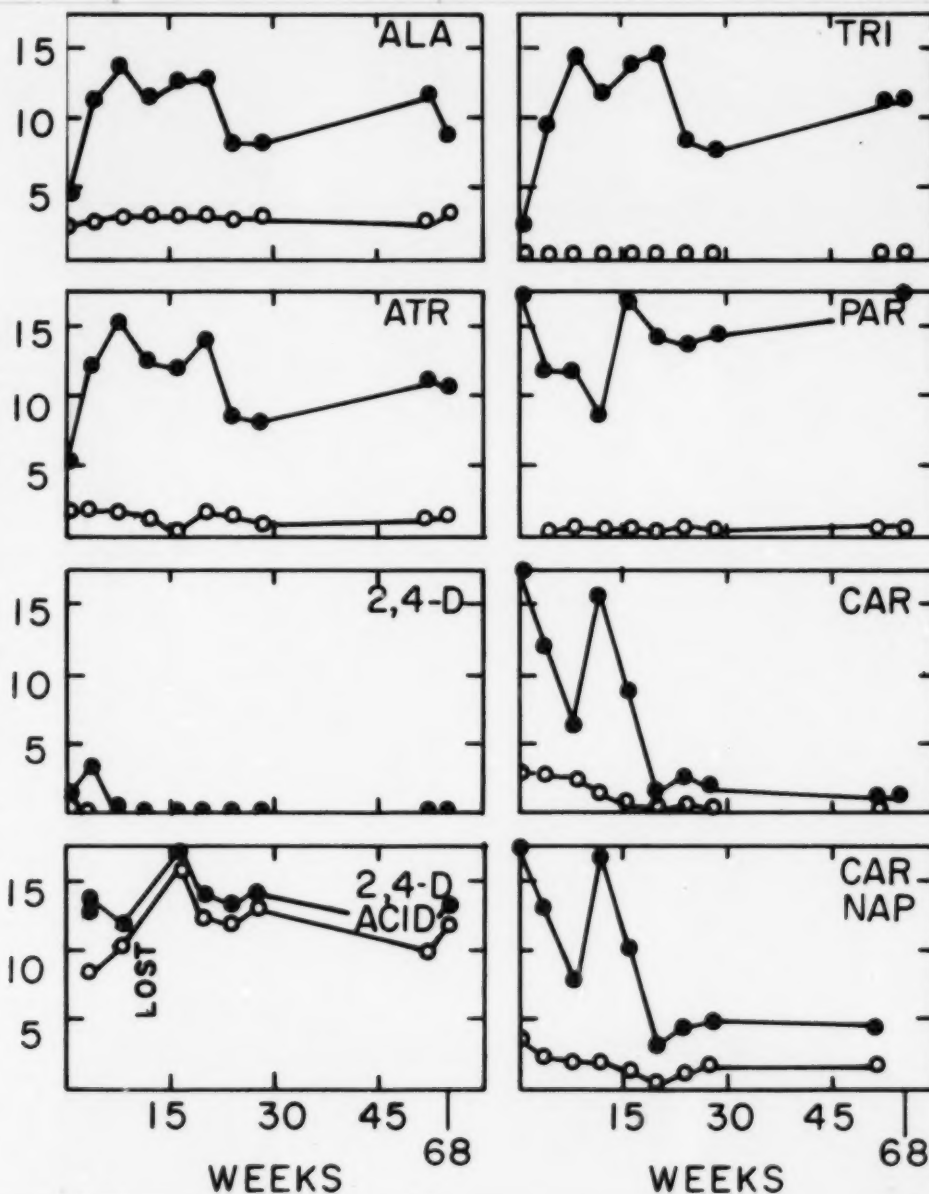


Figure 91. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15 Kg Soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, 0.1% peptone nutrient, and 5 other pesticides.

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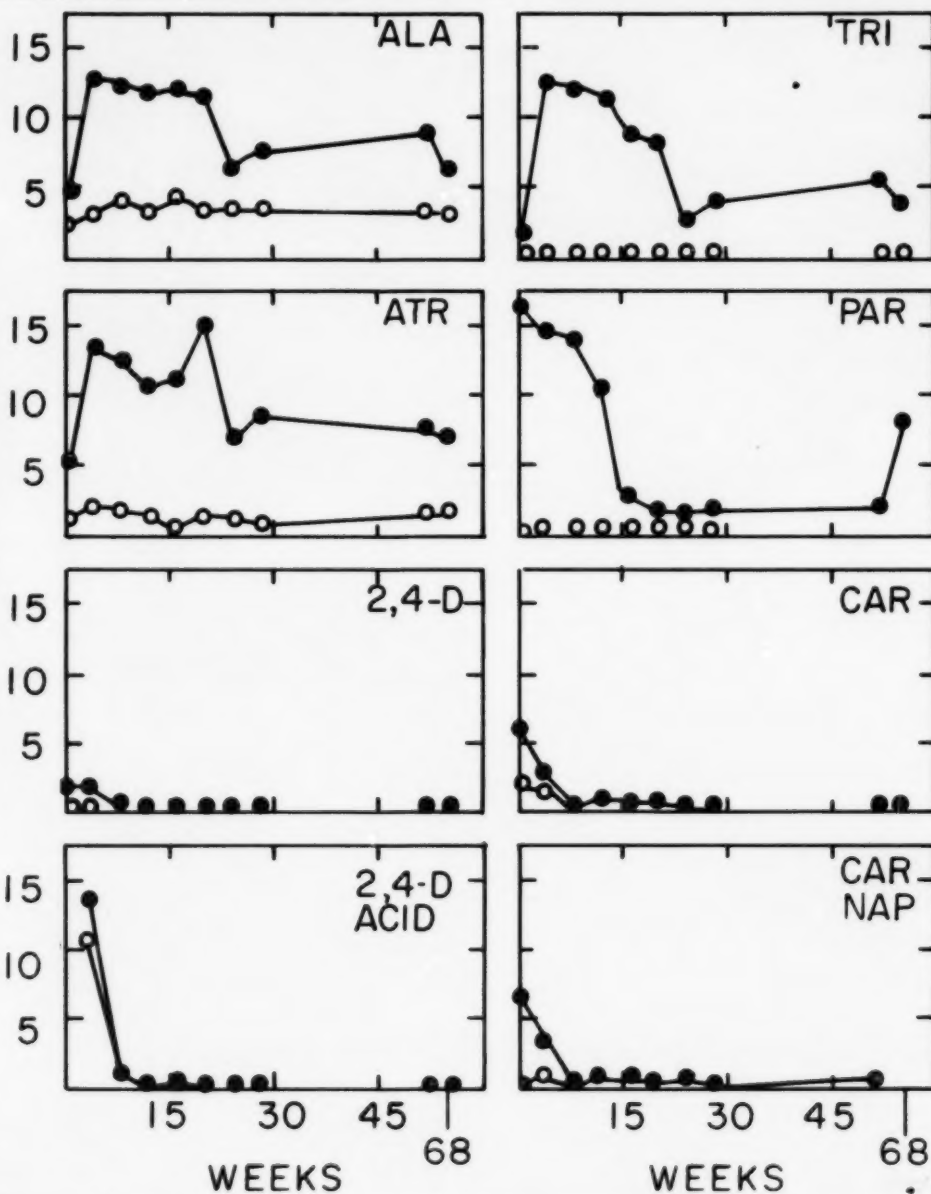


Figure 92. Decay from charge on 4/27/77 of 15 g of listed pesticide per 60 l H₂O and 15Kg Soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min air, 0.1% peptone nutrient, and 5 other pesticides.

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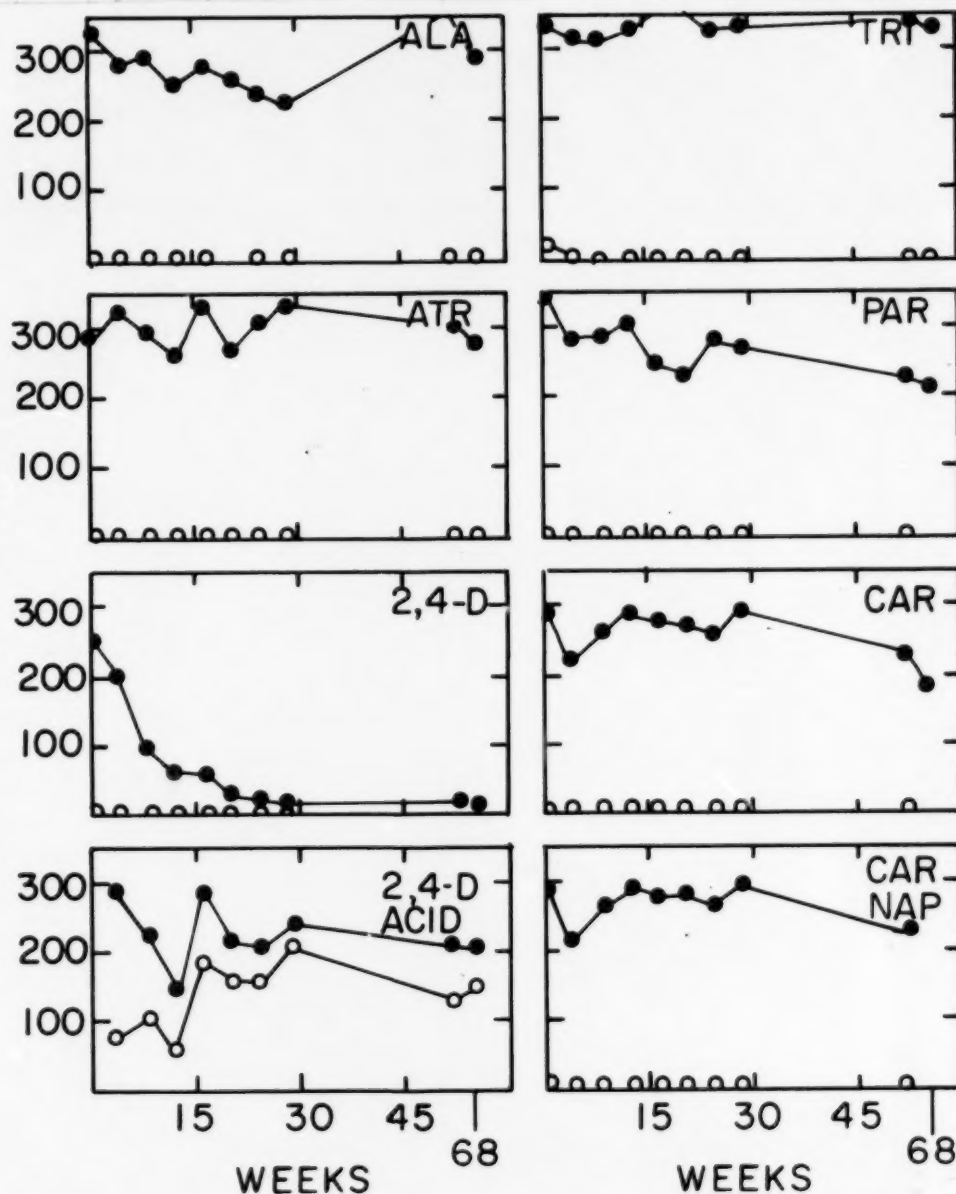


Figure 93. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg Soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, no nutrients, and no other pesticides.

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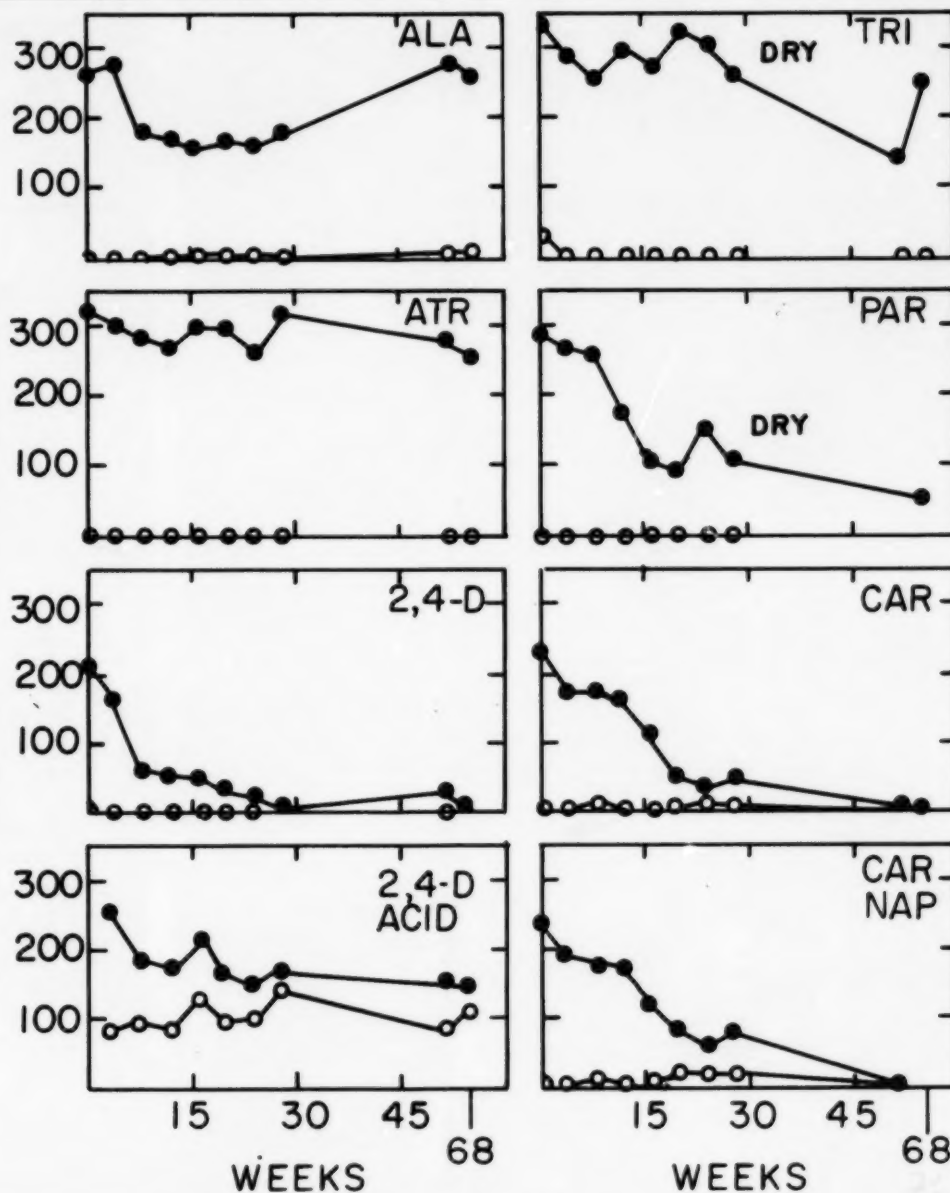


Figure 94. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min air, no nutrient, and no other pesticides.

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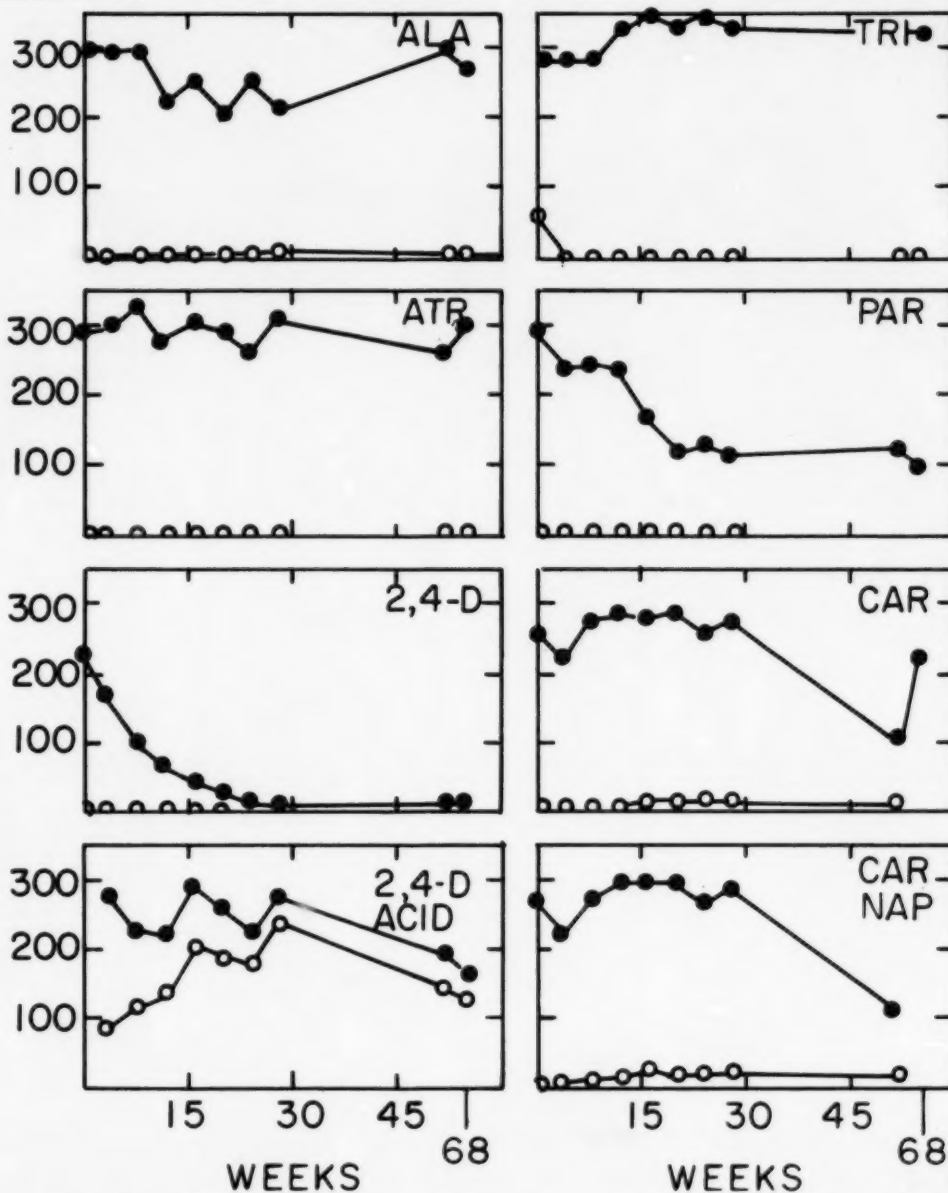


Figure 95. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, 0.1% peptone nutrient, and no other pesticides.

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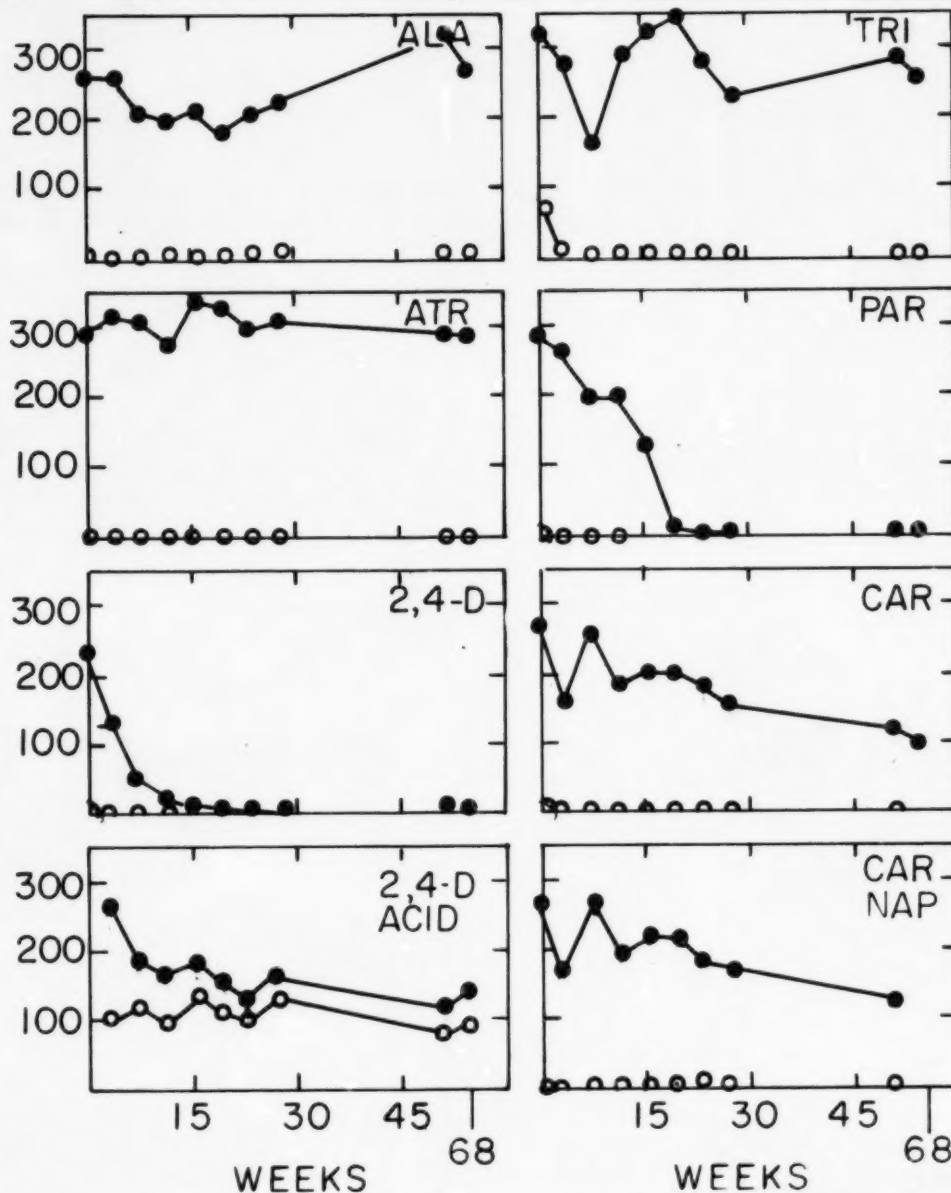


Figure 96. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min air, 0.1% peptone nutrient, and no other pesticides.

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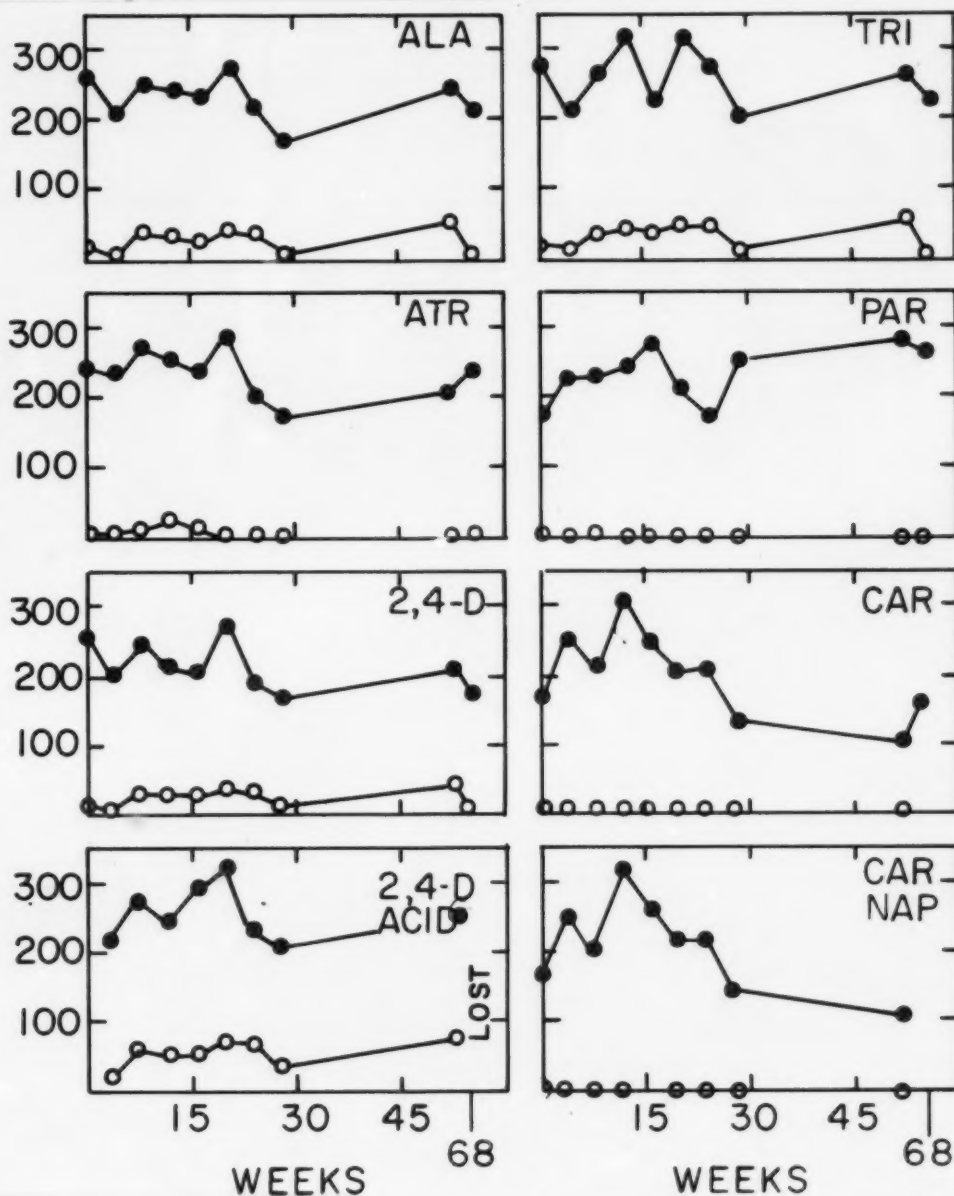


Figure 97. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = trifluralin; ATR = atrazine; PAR = parathion; 2,4-D = butoxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, no nutrient, and 5 other pesticides.

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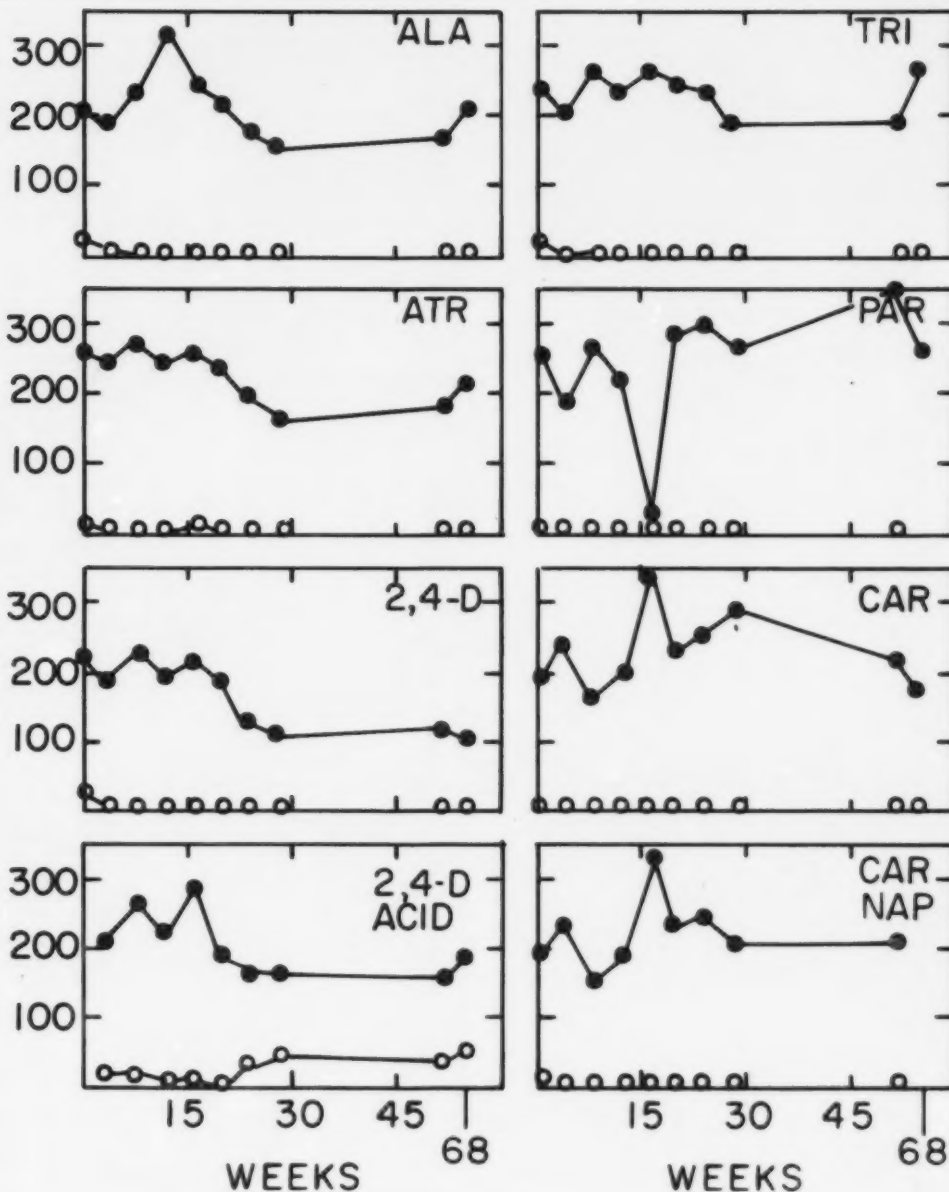


Figure 98. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min air, no nutrient, and 5 other pesticides.

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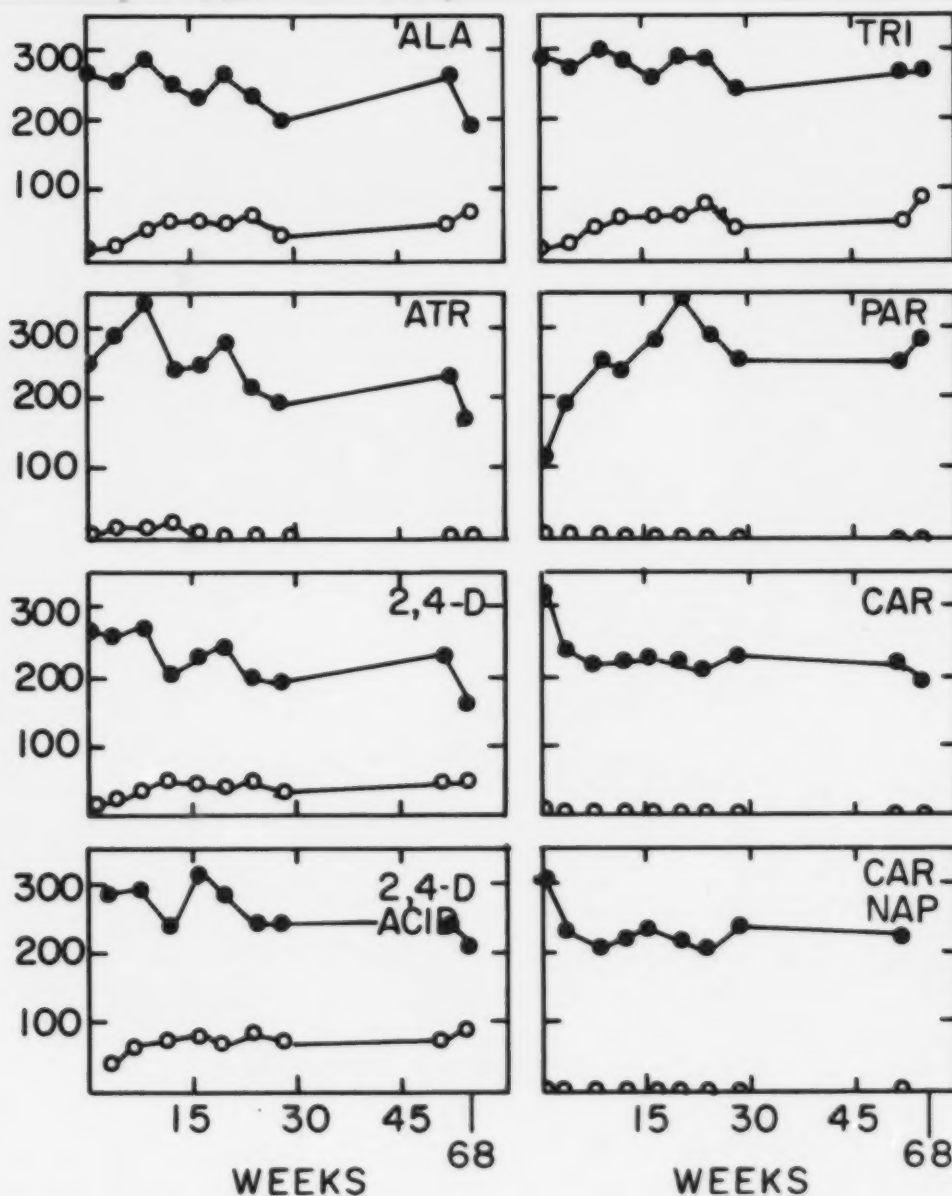


Figure 99. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlororphenoxycetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. No aeration, 0.1% peptone nutrient, and 5 other pesticides.

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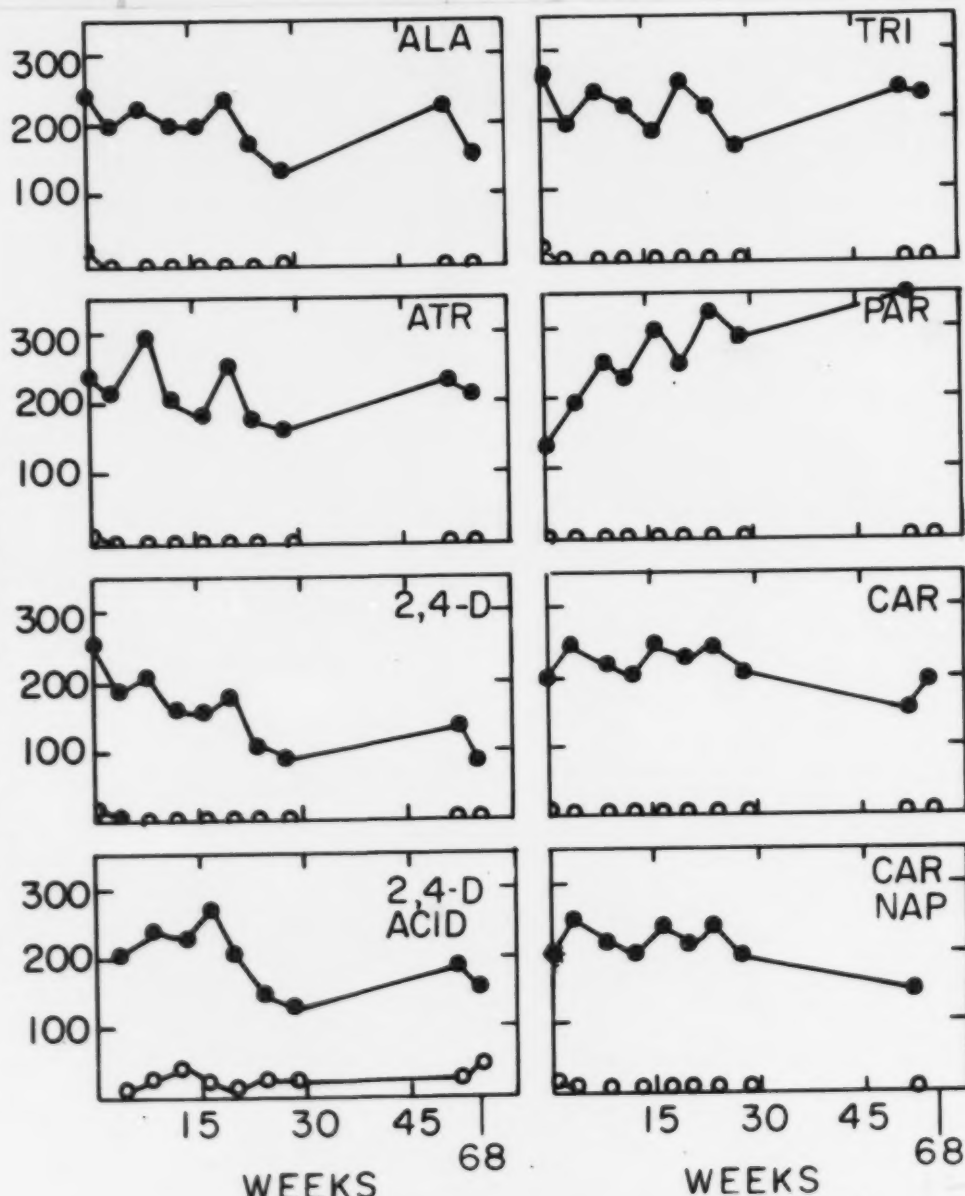


Figure 100. Decay from charge on 4/27/77 of 300 g of listed pesticide per 60 l H₂O and 15 Kg Soil (ALA = alachlor; TRI = tri-fluralin; ATR = atrazine; PAR = parathion; 2,4-D = but-oxyethanol ester of 2,4-dichlorophenoxyacetic acid; CAR = carbaryl; ACID = free acid of 2,4-D; NAP = 1-naphthol). ● = Amount in water and soil. ○ = Amount in water. ~1 l/min air, 0.1% peptone nutrient, and 5 other pesticides.

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TABLE 28 SUMMARY OF THE EFFECT OF AMBIENT CONDITIONS, AERATION AT 1 L/MIN, PEPTONE NUTRIENT AT 0.1 WT %, CONCENTRATION, AND THE PRESENCE OF 5 OTHER PESTICIDES (MIX) ON THE DECAY OF SIX PESTICIDES AND TWO DECOMPOSITION PRODUCTS IN MINI-PITS DESIGNED FOR PESTICIDE DISPOSAL.

Positive decay to <10% active ingredient within 68 weeks ^a																						
Condition	Initial amount in grams	Alachlor		Atrazine		2,4-D Ester		2,4-D Acid		Trifluralin		Parathion		Carbaryl		1-naphthol				Yes count [†]		
		Alone		Mix		Alone		Mix		Alone		Mix		Alone		Mix		Alone			Mix	
No nutrients: No aeration	15	NO	NO	NO	NO	YES	YES	YES	NO	YES?	NO	YES	NO	YES	YES	YES	YES	YES	YES	9		
	300	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	1		
Aeration	15	NO	NO	NO	NO	YES	YES	YES	YES	YES?	NO	YES	YES	YES	YES	YES	YES	YES	YES	11		
	300	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO	NO	YES	NO	YES	NO	YES	NO	3		
Nutrients: Aeration	15	NO	NO	NO	NO	YES	YES	YES	YES	YES	NO	YES	NO	YES	YES	YES	YES	YES	YES	10		
	300	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO	NO	2		
No aeration	15	YES?	NO	NO	NO	YES	YES	NO	NO	YES	NO	YES	NO	YES?	YES	YES	YES	YES	YES	8		
	300	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO	1		
Yes count ^g		1	0	0	0	8	4	4	3	2	4	5	1	5	4	5	3	8		45		

^aOnly 12 of which were conducive to decay because of drastic changes in seasonal temperature. Any borderline cases based on known analytical deviations are given a question mark following the YES for decay to <10% the initial amount. All pits were active microbiologically over the entire 68 week period.

^bA yes count of 8 indicate very easy degradability - a count of 0 indicate strong persistence. Mixture effect shown in column pairs.

^cA yes count of 16 would indicate complete degradability of all components at listed conditions - concentration effect shown in row pairs.

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even in the absence of either aeration or added nutrients. Many other deductions similar to this example can be made from inspection of these figures. However, the summation of these deductions results in a rather confusing representation of the overall effect which the variables, aeration, nutrients, mixtures, chemical structure, and concentration, have on the effectiveness of pit systems for the disposal of pesticides.

The influence of these variables can best be ascertained by consideration of the discussions in the section dealing with the Degradation Summary which follows immediately.

Degradation Summary--

The data plotted in Figures 69-84 were used to formulate the matrix summary shown in Table 28. For a generalized interpretation of the matrix, the six pesticides chosen for the minipit study are assumed to be representative of recalcitrant to easily degradable pesticides. The criteria for a positive (YES) degradation is decay to less than 10% of the amount originally deposited into the pit within the reasonable interval of 68 weeks. The reader should bear in mind that only about 30 of these 68 weeks are conducive to degradation for the seasonal climatic conditions where these pits were located in Iowa. A more quantitative matrix, which includes factors for the amount of decay at all tested intervals would not be very useful for interpretation purposes because of the possible bias related to the analytical uncertainties. These more subtle comparisons are best made by close inspection of the individual decay graphs in Figures 69-100 with due regard for the variation in analytical results.

The matrix summary presented here provides a very effective input for decisions regarding the addition of nutrients, the incorporation of aeration mechanisms, the change in the disposal load, the restriction on the dump of certain pesticides, etc. Some obvious observations about degradations from inspection of the matrix summary are presented in the following paragraphs.

The effects of all combinations of aeration and nutrients are easily discernable on the right hand YES count column. The increase in degradation from 10 to 14 decay units on a decay scale of 0 to 32 when aeration is incorporated must be weighed carefully against the cost of adding aeration to the pit system. Nutrients have an inhibitory, if any, effect on pesticide degradation in general.

Concentration has a dramatic effect on decay but the actual amount degraded per unit time must await future tests after longer periods of warm weather. For the present the effect of increased concentration by a factor of 20 (15 to 300 g) on a decay scale of 0 to 64 is reflected by the 38 decay units for low concentration (15 g) compared to only 7 for high concentration (300 g).

Mixtures and the associated increase in concentration have an inhibitory effect on degradation in every case with the most dramatic being the decays of 2,4-D ester, trifluralin and parathion. On a decay scale of 0 to 64, the individual pesticide degradation decreases from 31 to 14 decay units

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when mixtures are present. This apparent antagonistic action of mixtures may be more of a latency and concentration effect rather than a toxicity effect on microorganisms.

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For degradations in separate pits and a decay scale of 0 to 8, atrazine (0) is the most persistent pesticide followed in order by alachlor (1), 2,4-D acid (3), trifluralin (4), carbaryl (5), 1-naphthol (5), parathion (5), and 2,4-D ester (8).

This degradation order also exists when mixtures and individual pesticides are considered together on a degradation scale of 0 to 16 decay units.

For degradation when mixtures are present and a decay scale of 0 to 8, atrazine (0), alachlor (0), and trifluralin (0) are all persistent, followed in order by parathion (1), 2,4-D acid (2), 1-naphthol (3), carbaryl (4), and 2,4-D ester (4).

Other less important observations can be made by breaking down the relative degradations into pairs of low and high concentrations using decay scales of 0 to 4 decay units.

The seven important degradation observations discussed above can be supplemented by observations and conclusions made from the experience gained in the operation of the minipits and from other data accumulated during the course of these minipit studies. Interpretation of data from the study of several lab bench disposal systems also adds to the list of supplemental observations. These are itemized in the indented paragraphs which follow.

The rather large uncertainties in the analytical data, reflected by the decay graphs in Figures 69-100 are due primarily to sample homogeneity problems similar to those documented regularly in food analyses. Bench scale tests of soil, H₂O, and formulation mixtures indicate that more vigorous mixing prior to sampling would not be very beneficial. These analytical uncertainties should be considered carefully in making conclusions about decay rate and the effects of aeration, nutrients, mixtures, concentrations, etc.

Cool weather influences and cold weather interrupts the decay of pesticides as evidenced by the decay graphs in Figures 69-100. The temperature of the contents of the partially buried containers follows closely the seasonal changes.

No serious interruption in the decay process occurs when the same formulations are added to existing minipit systems (see trifluralin and 2,4-D ester respire data in Figures 73, 75, and 77). This may also apply to the addition of other formulations of trifluralin and 2,4-D ester, and other pesticides.

Only an insignificant loss of pesticides occurs when the liquid contents of the minipits are lost due to ruptures caused by the freeze-thaw cycles (see Figures 69-95) where the placement of the word DRY

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indicates the time of the rupture). Almost complete containment is achieved by transferring the solid contents to a new plastic container and adding fresh water. However, a more suitable container may be available to solve the rupture problem.

The water data plotted in Figures 69-100 show that most of the pesticides are sorbed on soil particles. This confirms what is probably the most beneficial effect of soil in the pits. It guarantees almost complete containment even when loss of the liquid contents occurs.

Buffers have not aided the degradation of large amounts of 2,4-D ester to products beyond the 2,4-D acid hydrolysis product.

No measurable losses due to purging of the pesticides occurs during aeration. The surprising lack of a purging effect may be due to the closed lid system.

Water Loss, pH and Temperature

On each of the days when samples were taken for analyses, the evaporation losses and the pH of the mixed pit contents were measured. On occasion the temperature of the pit contents was measured.

The evaporative losses in liters for each disposal pit are recorded in Tables 29-35. The losses since the previous sampling time and total accumulated losses up to 123 weeks are recorded. In general, the greatest losses occurred during the hot summer months. There is fair consistency between the losses for each of the pits when one considers the probable variations in the fit of the container lids and other factors such as channeled wind currents which can effect the evaporation. This consistency is reviewed here only for the data after 123 weeks for seven of the pits containing alachlor. If one refers to the far right-hand column in Table 29, the total losses of 27.9, 28.9, 25.5, 20.3, 27.1, 24.6, and 20.6 liters over a 123 week period are recorded. This is somewhat representative of the agreement in the water losses.

Whenever a container was ruptured, no further records of water losses are recorded in Tables 29-35 since obviously these losses were other than evaporative. The approximate surface area for the evaporation from these minipits was 0.4 m² but the lids were always in place except for sampling. A change to an open cover design would enhance the water evaporation and improve the possibility of photolytic degradations. With the systems tested here the closed lid approach gave evaporative losses of 12 to 16 liters per year. This loss may be determined from Figure 101 where the numerical data in Tables 29-35 have been averaged and plotted. In continuously hot climates with temperatures of 90-100°F, the water evaporation losses from similar closed lid systems would be about 2 l/month or 24 l/year. This figure is important since it represents the maximum volume of liquid waste which one of these pits can handle per month or per year.

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The comparable losses with and without aeration, as noted in the footnote in Figure 101, is quite surprising. There would probably be a difference

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TABLE 29. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH ALACHLOR ON 4/27/77*

Mini- pit	Liters Lost During Week Intervals												68-123 0-123
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	40-44	44-48	
1A	1.6	1.0	1.3	1.3	1.0	1.0	1.0	0.7	4.0	12.9	15.0	27.9	
2A	1.8	0.8	2.0	1.7	1.6	0.8	0.6	2.6	5.0	12.0	28.9		
3A	1.7	1.8	2.5	2.0	0.7	0.6	0.5	1.7	3.0	11.0	25.5		
4A	1.5	1.1	2.0	1.4	1.6	1.5	1.0	1.1	5.9	17.1	†		
5A	2.2	1.5	2.0	2.7	0.8	0.5	0.5	0.2	2.9	7.0	20.3		
6A	1.8	1.8	2.0	3.3	0.9	1.0	0.5	0.9	3.9	11.0	27.1		
7A	1.6	1.4	1.5	1.2	1.1	0.8	1.0	0.6	4.4	11.0	24.6		
8A	1.5	1.0	1.5	0.9	0.9	0.6	0.8	1.0	4.4	8.0	20.6		

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of Alachlor as Lasso®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 30. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH ATRAZINE ON 4/27/77*

Mini-pit	Liters Lost During Week Intervals										68-123 0-123
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	
1B	1.5	1.2	1.8	1.9	1.9	1.0	0.8	3.6	4.7	11.0	68-123 0-123
2B	1.7	0.8	1.0	1.8	1.8	1.8	0.6	3.4	14.0	†	29.4
3B	0.7	0.8	1.0	0.5	0.8	0.8	0.5	2.9	5.6	†	—
4B	1.7	0.4	1.0	2.3	1.4	1.5	0.8	2.9	5.0	12.0	29.0
5B	1.5	2.0	2.5	1.3	0.8	1.0	0.6	2.6	5.3	8.0	25.6
6B	2.0	1.4	2.0	1.7	0.7	0.8	0.5	3.6	4.0	11.0	27.7
7B	1.5	1.2	1.5	1.3	0.7	0.8	0.7	2.9	5.4	10.0	26.0
8B	0.6	1.0	2.3	1.6	1.0	0.8	1.0	2.7	4.3	10.0	25.4

* The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of Atrazine as Aatrex®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 31. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH 2,4-D BUTOXYETHANOL ESTER ON 4/27/77*

Mini- pit	Liters Lost During Week Intervals														
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	40-44	44-48	48-52	52-56	
1C	1.6	1.2	2.8	4.5	6.0	7.2	8.1	8.9	11.9	16.8	11.0	27.8	68-123	0-123	
2C	1.6	1.2	1.3	2.2	6.3	7.2	8.8	10.4	13.4	17.2	†	—			
3C	1.7	1.5	1.8	2.2	7.2	8.1	9.0	9.7	12.9	16.8	†	—			
4C	1.5	1.2	1.5	2.0	6.2	7.7	9.3	10.1	13.6	16.4	9.0	25.4			
5C	1.4	1.0	2.0	1.5	5.9	7.3	8.0	9.6	12.7	16.3	9.0	25.3			
6C	1.7	1.4	1.5	1.9	6.5	8.2	9.0	10.0	14.2	17.9	13.0	30.9			
7C	1.9	1.4	2.5	2.2	8.0	9.0	10.4	11.2	14.4	19.2	10.0	29.2			
8C	1.9	1.6	1.5	2.0	7.0	7.8	9.3	10.0	—	—	—	—			

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of 2,4-D butoxyethanol ester as 2,4-D as Weedone®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 32. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH TRIFLURALIN ON 4/27/77*

Mini-pit	Liters Lost During Week Intervals																			68-123 0-123
	0-4 0-4	4-8 0-8	8-12 0-12	12-16 0-16	16-20 0-20	20-24 0-24	24-28 0-28	28-52 0-52	52-68 0-68											
1D	2.0 2.0	1.2 2.2	2.5 4.5	1.3 5.8	1.2 7.0	1.1 8.1	0.7 8.8	4.0 12.8	5.0 17.8	†										
2D	1.6 1.6	1.2 2.8	1.5 4.3	1.8 6.1	0.9 7.0	1.7 8.7	0.4 9.1	16.5 25.6	4.9 30.5	10.0 40.5										
3D	2.4 2.4	1.9 4.3	3.0 7.3	2.4 9.7	1.2 11.2	0.9 12.1	1.0 13.1	3.1 16.2	4.5 20.7	11.0 31.7										
4D	1.5 1.5	1.0 2.5	0.5 3.0	1.2 4.2	0.7 4.9	0.5 5.4	0.5 5.9	2.1 8.0	4.0 12.0	†										
5D	2.2 2.2	1.0 3.2	3.0 6.2	2.7 8.9	1.0 9.9	0.6 10.5	1.0 11.5	†	—	—										
6D	1.6 1.6	0.8 2.4	2.0 4.4	0.8 5.2	1.3 6.5	0.7 7.2	0.5 7.7	2.8 10.5	4.7 15.2	†										
7D	1.9 1.9	0.6 2.5	1.5 4.0	0.9 4.9	0.9 5.8	0.6 6.4	0.4 6.8	2.9 9.7	4.0 13.7	10.0 23.7										
8D	1.6 1.6	1.1 2.7	2.0 4.7	1.5 6.2	0.8 7.0	0.8 7.8	0.6 8.4	†	—	—										

* The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of Trifluralin as Treflan®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 33. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH CARBARYL ON 4/27/77*

Mini- pit	Liters Lost During Week Intervals											
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	40-44	44-48
1E	1.4	1.8	1.8	1.0	1.4	1.7	0.4	3.0	12.5	2.5	15.0	†
2E	1.2	1.3	1.3	1.8	1.5	1.8	0.8	3.1	12.8	4.0	12.0	28.8
3E	1.2	1.2	1.3	1.4	1.0	1.4	1.3	6.4	15.2	3.6	†	—
4E	1.4	1.1	1.8	1.5	1.7	1.2	0.9	2.2	11.8	5.1	11.0	27.9
5E	1.3	1.4	1.5	0.7	1.1	1.2	0.4	†	—	—	—	—
6E	1.6	1.6	1.8	1.5	1.4	1.6	1.1	†	—	—	—	—
7E	1.1	1.6	2.5	2.1	0.9	1.6	0.3	3.4	13.5	4.9	11.0	29.4
8E	1.8	1.4	2.5	2.4	1.0	0.0	0.0	†	—	—	—	—

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of Carbaryl as Sevin®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

†Ruptured container and all the liquid was lost.

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TABLE 34. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH PARATHION ON 4/27/77*

Mini- pit	Liters Lost During Week Intervals												
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	40-44	44-48	
1F	1.6	1.3	2.0	2.3	1.0	1.4	1.1	2.5	2.7	13.0	13.0	28.9	68-123
	1.6	2.9	4.9	7.2	8.2	9.6	10.7	13.2	15.9	15.9	15.9	15.9	0-123
2F	1.0	1.3	1.6	1.9	2.4	1.4	0.8	3.3	3.9	13.0	13.0	30.6	
	1.0	2.3	3.9	5.8	8.2	9.6	10.4	13.7	17.6	17.6	17.6	17.6	
3F	1.5	1.5	1.8	1.4	1.0	1.5	0.9	2.0	4.2	12.0	12.0	27.8	
	1.5	3.0	4.8	6.2	7.2	8.7	9.6	11.6	15.8	15.8	15.8	15.8	
4F	1.3	2.0	1.7	2.5	1.0	1.4	0.4	2.9	3.9	11.0	11.0	28.1	
	1.3	3.3	5.0	7.5	8.5	9.9	10.3	13.2	17.1	17.1	17.1	17.1	
5F	1.8	1.9	2.3	1.4	1.0	0.9	1.0	1.0	—	—	—	—	
	1.8	3.7	6.0	7.4	8.4	9.3	10.3	—	—	—	—	—	
6F	1.6	1.7	1.6	2.2	1.6	1.1	0.0	1.3	4.0	11.0	11.0	26.1	
	1.6	3.3	4.9	7.1	8.7	9.8	9.8	11.1	15.1	15.1	15.1	15.1	
7F	1.4	1.7	2.4	2.7	0.9	2.0	0.8	1.6	4.4	12.0	12.0	29.9	
	1.4	3.1	5.5	8.2	9.1	11.1	11.9	13.5	17.9	17.9	17.9	17.9	
8F	1.3	1.2	2.0	2.4	0.9	1.9	0.0	0.0	3.0	—	—	—	
	1.3	2.5	4.5	6.9	7.8	9.7	9.7	9.7	12.7	12.7	12.7	12.7	

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of Parathion as Security®; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 35. WATER EVAPORATION FROM MINI-PITS SINCE CHARGING WITH ALACHLOR, ATRAZINE, 2,4-D BUTOXYETHANOL ESTER, TRIFLURALIN, CARBARYL, AND PARATHION ON 4/27/77*

Mini-pit	Liters Lost During Week Intervals												68-123
	0-4	4-8	8-12	12-16	16-20	20-24	24-28	28-32	32-36	36-40	40-44	44-48	
1G	2.7	2.5	2.0	2.7	1.4	0.9	1.0	3.5	5.7	14.0	14.0	14.0	14.0
2G	2.7	5.2	7.2	9.9	11.3	12.2	13.2	16.7	22.4	36.4	36.4	36.4	36.4
3G	1.9	2.3	2.0	2.9	1.3	1.3	1.6	3.4	5.7	14.0	14.0	14.0	14.0
4G	1.9	4.2	6.2	9.1	10.4	11.7	13.3	16.7	22.4	36.4	36.4	36.4	36.4
5G	2.4	2.3	1.0	2.4	0.4	1.5	1.4	2.4	5.8	13.0	13.0	13.0	13.0
6G	2.4	4.7	5.7	8.1	8.5	10.0	11.4	13.8	19.6	32.6	32.6	32.6	32.6
7G	1.6	1.4	1.4	1.3	1.0	0.7	1.0	2.7	4.0	14.0	14.0	14.0	14.0
8G	1.6	3.0	4.4	5.7	6.7	7.4	8.4	11.1	15.1	29.1	29.1	29.1	29.1
9G	1.6	1.6	0.8	1.0	0.4	1.2	0.9	0.4	3.2	†	†	†	†
10G	1.6	3.2	4.0	5.0	5.4	6.6	7.5	7.9	11.1	—	—	—	—
11G	1.9	1.4	1.1	2.8	1.1	1.6	0.8	3.4	5.0	12.0	12.0	12.0	12.0
12G	1.9	3.3	4.4	7.2	8.3	9.9	10.7	14.1	19.1	31.1	31.1	31.1	31.1
13G	1.8	1.7	1.3	3.0	1.5	1.4	1.0	4.0	5.0	10.0	10.0	10.0	10.0
14G	1.8	3.5	4.8	7.8	9.3	10.7	11.7	15.7	20.7	30.7	30.7	30.7	30.7
15G	1.7	1.7	3.0	2.0	1.4	1.7	1.0	3.3	4.3	10.0	10.0	10.0	10.0
16G	1.7	3.4	6.4	8.4	9.8	11.5	12.5	15.8	20.1	30.1	30.1	30.1	30.1

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic container. All even numbers have 15 g of the active ingredient of each pesticide formulation; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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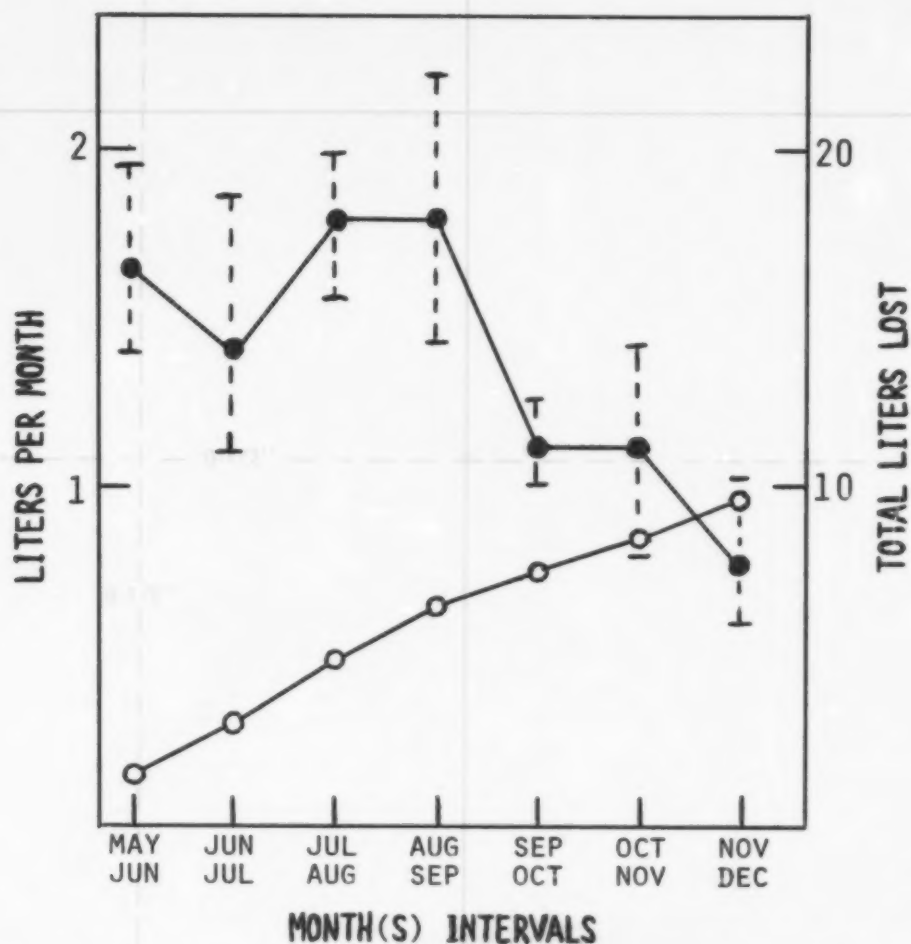
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Figure 101. Average loss per month (●) and average total H₂O Loss (○) During 1977 for 56 mini-pits, 28 of which were aerated at 1 l/min. (No difference observed for aerated versus non-aerated pits. Brackets indicate the maximum and minimum loss at each monthly interval. The H₂O losses for 1978 and 1979 were the same as shown here for 1977.)

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in these two kinds of systems if an open lid design were used in a continuously hot region.

The pH measurements were made immediately after taking the soil and liquid samples. No doubt, the presence of a very large amount of suspended sediment affected the accuracy and reproducibility of these pH measurements. The measured values for each pit are recorded in Tables 36-42. The deposited pesticides, the formulating agents and the decomposition products did not change the pH from that observed for ambient water except for the minipits 1C, 3C, 5C and 7C where high concentrations of 2,4-D butoxyethanol as Weedone were deposited. The analytical data for these pits show that rapid hydrolysis to the acid occurs. It is this acid which causes the low pH. However even this pH is not too low for survival of most all microorganisms. Indeed the counts of microorganisms from these particular pits, as discussed in the section of Microbiological Studies, are no different than the other pits. A comparable amount of Weedone[®] was placed in the pits listed in Table 42 but lowering of the pH did not occur here. In some cases this is due to slower decay when a mixture is present but even when the analytical data show that the ester has been hydrolyzed, no pH near 5 is observed. Obviously, one of the substances used in one of the other five formulations acts as a buffer.

All of our data suggest that pH of the pit system is of concern only if large amounts of acidic pesticides or esters which are easily hydrolyzed are being dumped. This is not to say that pH is unimportant for some degradations. The pH may well be a consideration of a particular pesticide decay but it seems to be unimportant for a general disposal system. Nevertheless the pH of any pit system should be checked regularly because it can be done so easily. If necessary then, buffers can be added if the pH becomes low enough to affect the microorganisms or the degradation of some chemicals of concern.

The temperature of the pits was not measured regularly. In general the ground acts as a buffer for short term and seasonal changes in temperature. For example, the pits still have ice in them in April long after the ground has thawed. On the other end of the temperature scale, the pits are still liquid in November long after stagnant surface waters have frozen. In mid-summer, the pit temperatures are stable at about 22°C.

Laboratory Decay Studies

Most of the laboratory decay studies were to be completed during the last two years, 1980 and 1981, of the projected five year study, 1977 to 1981. These experiments were terminated when funding for the final two years was not granted. However some bench-scale degradation experiments were completed and the data from the study of the decay of 2,4-D butoxyethanol ester, alachlor, treflan and atrazine under a variety of aerobic and anaerobic conditions are tabulated in Tables 43 and 44.

The review of the data for these bench scale studies can be used for the following preliminary observations: 1) ambient soil is about as good an inoculant as are the aerobic and anaerobic slimes and sludges; 2) artificial

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TABLE 36. pH VALUES OF MINI-PITS CHARGED WITH ALACHLOR AS LASSO® ON 4/27/77*

Mini-pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1A	6.4	6.6	6.5	6.8	6.8	6.8	6.9	6.5	6.9
2A	6.4	6.8	6.6	7.2	7.2	7.2	7.2	6.7	7.1
3A	6.8	7.2	7.4	7.1	7.3	7.3	7.2	6.7	†
4A	6.9	7.0	6.8	6.8	7.2	7.2	7.3	6.7	7.1
5A	6.4	6.6	6.5	6.5	7.1	6.8	6.9	6.6	7.0
6A	6.7	6.6	6.6	6.6	7.0	6.9	7.2	6.7	7.0
7A	6.4	6.6	6.7	6.5	6.9	6.8	6.4	6.7	6.9
8A	6.5	6.6	6.5	6.6	6.8	6.8	7.1	6.7	6.9

TABLE 37. pH VALUES OF MINI-PITS CHARGED WITH ATRAZINE AS AATREX® ON 4/27/77*

Mini-pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1B	6.9	7.0	7.0	7.1	7.4	7.4	7.2	6.7	7.0
2B	7.0	7.0	6.9	6.8	7.1	7.3	7.4	6.5	†
3B	6.8	7.0	6.9	6.8	7.1	7.1	7.4	6.4	†
4B	7.0	6.9	6.7	6.8	7.1	7.1	7.1	6.5	7.0
5B	6.4	6.7	6.8	6.7	7.1	7.0	7.0	6.5	7.0
6B	6.3	6.7	6.8	6.8	7.1	7.1	7.1	6.5	7.0
7B	6.5	6.8	6.8	6.8	7.0	7.2	7.1	6.7	6.9
8B	6.6	6.6	6.7	6.9	6.8	7.1	7.2	6.6	6.8

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic garbage container. All even numbers have 15 g of the active ingredient. All odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 38. pH VALUES OF MINI-PITS CHARGED WITH
2,4-D BUTOXYETHANOL ESTER AS WEEDONE® ON 4/27/77*

Mini- pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1C	5.4	4.9	5.0	4.9	4.8	4.8	5.2	5.5	5.0
2C	6.9	6.9	6.9	7.1	7.3	7.3	7.4	6.5	†
3C	4.7	4.3	4.2	4.3	4.2	4.3	4.9	4.9	†
4C	7.1	6.8	6.7	6.7	6.6	7.3	7.1	6.2	6.8
5C	4.6	4.3	4.2	4.1	4.2	4.2	4.3	4.7	4.2
6C	6.6	6.7	6.6	6.6	6.7	7.3	6.8	6.2	6.5
7C	5.0	4.7	4.6	4.6	4.5	4.5	4.9	5.2	5.9
8C	6.5	6.4	6.5	6.6	6.8	7.1	†	6.4	6.7

TABLE 39. pH VALUES OF MINI-PITS CHARGED WITH
TRIFLURALIN AS TREFLAN® ON 4/27/77*

Mini- pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1D	6.7	6.7	6.6	6.6	6.8	6.8	6.9	6.4	†
2D	6.9	6.8	6.8	6.8	7.2	7.1	7.1	6.7	7.2
3D	6.9	6.8	6.8	6.5	7.2	7.3	6.9	6.7	7.0
4D	6.9	6.9	6.8	6.5	7.2	7.1	6.9	6.5	†
5D	6.7	6.6	6.5	6.3	7.2	6.8	†	6.8	7.1
6D	6.8	6.7	6.6	6.5	7.0	6.9	6.8	6.4	†
7D	6.6	6.6	6.5	6.3	7.0	6.5	6.5	6.7	7.0
8D	6.7	6.7	6.7	6.5	6.9	6.5	†	6.6	7.1

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic garbage container. All even numbers have 15 g of the active ingredient. All odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

†Ruptured container and all the liquid was lost.

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TABLE 40. pH VALUES OF MINI-PITS CHARGED WITH
CARBARYL AS SEVIN® ON 4/27/77*

Mini- pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1E	6.7	7.1	7.2	7.2	7.3	6.9	7.3	6.3	†
2E	6.7	7.2	7.3	7.2	7.4	7.1	7.4	6.6	7.0
3E	7.0	7.4	7.5	7.4	7.8	7.5	7.4	7.0	†
4E	6.9	7.2	7.1	6.8	6.9	7.4	7.0	6.6	7.1
5E	7.1	7.4	7.5	7.6	7.7	7.7	†	7.1	7.0
6E	6.9	7.0	7.2	7.2	6.9	7.2	†	6.8	7.2
7E	6.9	7.0	7.1	7.2	7.1	7.2	7.1	6.8	7.1
8E	6.9	7.1	7.1	7.1	7.0	7.3	†	6.6	7.2

TABLE 41. pH VALUES OF MINI-PITS CHARGED WITH
PARATHION AS SECURITY® ON 4/27/77*

Mini- pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1F	6.9	7.0	7.1	7.0	7.0	7.1	6.9	6.6	6.8
2F	6.9	7.1	7.3	7.3	7.4	7.5	7.2	6.6	6.9
3F	7.0	7.2	7.4	7.3	6.5	7.1	6.7	6.3	6.4
4F	7.1	7.5	7.4	7.1	7.3	7.3	7.2	7.2	6.8
5F	7.1	6.7	6.4	6.4	6.4	7.0	†	6.3	†
6F	7.1	6.9	7.0	7.2	6.8	7.3	7.0	6.5	7.1
7F	7.0	7.0	7.0	7.0	7.0	7.3	6.9	6.7	6.8
8F	7.2	6.9	6.9	7.1	6.9	7.4	6.9	6.6	†

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic garbage container. All even numbers have 15 g of the active ingredient. All odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 42. pH VALUES OF MINI-PITS CHARGED WITH
ALACHLOR, ATRAZINE, 2,4-D BUTOXYETHANOL ESTER,
TRIFLURALIN, CARBARYL AND PARATHION ON 4/27/77*

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Mini- pit	pH at weeks since charging								
	8	12	16	20	24	28	52	68	123
1G	6.8	6.7	6.9	6.8	6.9	6.8	6.8	6.6	6.9
2G	7.1	6.9	7.2	6.8	7.1	7.1	6.9	6.8	7.2
3G	6.9	6.7	6.3	6.2	6.4	6.1	6.4	6.5	6.6
4G	7.2	7.0	7.1	6.6	7.0	6.8	7.0	6.5	6.8
5G	7.0	6.4	6.6	6.5	6.5	6.3	6.0	6.4	†
6G	7.0	6.7	6.8	6.6	6.9	6.8	6.7	6.5	6.9
7G	6.9	6.7	6.9	6.6	6.8	6.6	6.7	6.5	6.7
8G	6.9	6.7	6.9	6.6	6.9	6.6	6.6	6.6	6.8

*The initial charge of each mini-pit was 15 kg soil and 60 l H₂O to a 125 l plastic garbage container. All even numbers have 15 g of the active ingredient of each pesticide formulation; all odd numbers have 300 g; all 1-4 mini-pits contain 0.1% peptone nutrient; all 3-6 mini-pits have 1 l/min. aeration.

† Ruptured container and all the liquid was lost.

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TABLE 43. AMOUNTS OF HERBICIDES RECOVERED FROM SMALL AEROBIC AND ANAEROBIC DIGESTERS CONTAINING 500 ml H₂O AND VARIOUS COMBINATIONS OF DIRT, PEPTONE, SEWAGE SLUDGE AND AEROBIC SLIME AS LISTED UNDER CONDITIONS

Grams	Pesticide Conditions	Grams Remaining After				
		0-wks	7-wks	13-wks	21-wks	35-wks
1.8	2,4-D Ester*					
	Anaerobic					
	dirt, solid sludge	1.8	1.4	1.5	1.5	1.6
	dirt	2.4	1.4	1.6	1.5	1.8
	peptone, solid sludge	1.9	2.0	2.2	2.0	1.9
1.8	2,4-D Ester					
	Aerobic					
	dirt, slime	2.0	1.3	1.5	1.5	1.5
	dirt	2.0	1.7	1.7	1.5	1.7
	peptone, slime	2.0	1.5	1.5	1.5	1.8
0.1	Alachlor					
	Anaerobic					
	dirt, solid sludge	0.1	0.1	0.1	0.1	0.1
	dirt	0.1	0.1	0.1	0.1	0.1
	peptone, solid sludge	0.1	0.1	0.1	0.1	0.1
0.1	Alachlor					
	Aerobic					
	dirt, slime	0.1	0.1	0.1	0.1	0.0
	dirt	0.1	0.1	0.1	0.1	0.1
	peptone, slime	0.1	0.0	0.0	0.0	0.0
0.1	Treflan					
	Anaerobic					
	dirt, solid sludge	0.1	0.1	0.0	0.0	0.0
	dirt	0.1	0.2	0.0	0.0	0.0
	peptone, solid sludge	0.1	0.1	0.0	0.0	0.0

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Grams	Pesticide Conditions	Grams Remaining After				
		0-wks	7-wks	13-wks	21-wks	35-wks
0.1	Treflan					
	Aerobic					
	dirt, slime	0.1	0.0	0.0	0.0	0.0
	dirt	0.1	0.1	0.1	0.0	0.0
	peptone, slime	0.1	0.0	0.0	0.0	0.0

*NaHCO₃ buffered at 13 weeks

TABLE . AMOUNT OF ATRAZINE AS AATREX[®] RECOVERED FROM SMALL
AEROBIC DIGESTERS CONTAINING 500 ml H₂O AND SEWAGE SLUDGE
AS LISTED UNDER CONDITIONS

Grams	Conditions	Grams Remaining After			
		0-wks	7-wks	11-wks	20-wks
1.0	0.5 g solid sludge, light	0.9	0.7	0.7	0.6
1.0	0.5 g solid sludge, dark	0.9	0.7	0.6	0.7
1.0	1 ml liquid sludge, light	0.9	0.7	0.7	0.7
1.0	1 ml liquid sludge, dark	0.9	0.7	0.6	0.7
0.3	0.5 g solid sludge, light	0.3	0.3	0.2	0.2
0.3	0.5 g solid sludge, light, stirred	0.3	0.2	0.2	0.2
0.3	0.5 g solid sludge, dark	0.3	0.3	0.2	0.2
0.3	1 ml liquid sludge, light	0.3	0.2	0.2	0.2
0.3	1 ml liquid sludge, light, stirred	0.3	0.3	0.2	0.2
0.3	1 ml liquid sludge, dark	0.3	0.3	0.2	0.2

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nutrients are not very useful for degradation purposes; 3) mixing has a questionable value for decay enhancement; 4) photolytic effects are not measurable for the decay of atrazine which is recalcitrant under all conditions investigated.

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Additional experiments with high concentrations of 2,4-D butoxyethanol ester with and without buffers showed no difference in degradation rates which confirm the observations made in field experiments. Neither the data for these field experiments or the bench scale experiments are tabulated in this report.

Combinations of buffers, soil, anaerobic sludge, aerobic slime, light and constant mixing are not considered to be useful for small and intermediate sized disposal systems.

Evidence of Decay

In the experimental design used for the study of the macropits and the minipits, the evidence of decay is usually indirect in that only the deposited pesticide was monitored. When the amounts decreased with time, chemical and biological decay mechanisms were assumed because our evaporation and leakage studies showed that the losses could not be accounted for by these routes. However, some losses by routes which could not be detected by the analytical regime always remain a possibility. Thus, the best evidence of decay is the observation of known degradation products. If the concentration of the degradation product increases concurrent with a decrease in concentration of the parent pesticide then this direct evidence is even more convincing. However, such an increase is not essential to prove degradation and may never occur in some cases due to an equal rate of decay of the primary degradation product to a less complicated component.

Those degradation products which have been identified in our study of various disposal systems are 1-naphthol from carbaryl, 2,4-D acid and 2,4-dichlorophenol from 2,4-D ester, 2-chloro-2',6'-diethylacetanilide from alachlor, α,α,α -trifluoro-2-nitro-6-amino-N,N-dipropyl-p-toluidine and α,α,α -trifluoro-2,6-diamino-N,N-dipropyl-p-toluidine from trifluralin, tetrachloroterephthalic acid from dimethyl-2,3,5,6-tetrachloroterephthalate, various thiophosphoric acids from a variety of organophosphate insecticides and a variety of phenols and acids from certain pesticides and from the decay of the aromatic solvents used in the formulation of the liquid pesticides as emulsifiable concentrates. About 60 other decay products were also identified but these identifications have not yet been confirmed positively.

Segregation and Composite Sampling

The chemicals deposited in the Horticulture and the Agronomy macropits exhibited an extreme tendency toward segregation. The segregation applies to both solid and liquid samples. The heterogeneous nature of the chemical composition of these pits, due to this segregation, makes it very difficult to obtain a valid sample which is truly representative of the pit contents.

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Therefore any attempt to obtain an accurate mass balance for a system which contains soil, rock, water and the deposited pesticides is certain to fail.

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The observed segregation is predictable because pesticides are manufactured and formulated to adhere to soil particles so that the activity persists and the losses due to runoff and leaching are minimized. The obvious slow approach to an equilibrium distribution within the pit systems attests to the success of the formulations and pesticides in fulfilling the slow migration intent. This same difficulty in securing a valid sample because of adsorption on soil particles was even experienced in the collection of soil and liquid samples from the much smaller minipits where rocks were not present and it was possible to mix the entire contents of the pit prior to taking the sample.

The compositing of the soil and the water samples prior to extractions and analyses is valid. This saves much analysis time but one must be careful to interpret the results of composite samplings in the light of the probable segregation within the pit system to be tested for pesticide residues. In the absence of results for separate samples to warn the analyst of the difficulty of obtaining a representative sample, too much significance can be placed on the results of composite samplings.

Containment of Pesticides

Containment in the systems under investigation was adequate though not absolute. Some losses due to leakage, spillage and vaporization did occur but these total losses were negligible compared to the total amount of deposited pesticides and to that amount which was degraded by chemical and biological mechanisms. In addition, losses by these undesirable routes did not have an adverse effect on the surrounding air and water environments.

The value of soil for the containment of the pesticides in a pit disposal system is documented by the analytical results for all the pit systems which were investigated. These included macro, micro, mini and bench scale systems. Chemical data show that the soil decreases the losses due to volatilization from the liquid surface and due to leakage through cracks in the container walls. Even the fully solubilized 2,4-D acid did not escape containment when the liquid contents of the minipits were lost entirely due to a rupture in the bottom of the container such that the liquid had to filter through the soil column before being lost to the surrounding ground.

This documentation of containment can be used to ease the analytical effort for many pit disposal systems because the solid contents would never be lost short of a catastrophic earthquake. Therefore analyses of the liquid contents may be sufficient to determine losses due to leakage.

For some pesticides the vapor losses could be significant but no appreciable losses have been measured in this current study of pit disposal systems. An additional discussion of containment is in the Agronomy Macropits section presented earlier.

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Many of the conclusions based on interpretation of the chemical results and the experience gained in these studies of pesticide disposal systems are discussed in the Results above and in the Experimental Section. Some of the more important ones are summarized briefly in the following paragraphs.

The effectiveness of a pit system containing soil can be measured by regular chemical analyses of the liquid contents of the pit. The conclusion presupposes proper placement of the pit system in an area of minimum leaching possibility.

Chemical analyses of the soil is extremely difficult and the results are questionable due to sampling and extraction problems.

The presence of rock as part of the solid contents of the pit can create insurmountable analytical problems and no advantages of rock are documented by any of the chemical results.

The difficulty is getting a representative sample of the solid contents is common to analytical chemistry and applies in particular to the pit systems described in this report and to other environments such as land-fills and sediments which receive a changing and complex organic load.

Soil is a very effective adsorption and ion exchange material as well as a food and organism source.

Methods for extraction and analysis of ionic degradation and metabolic products are not nearly as satisfactory as those for the primary and usually neutral waste chemicals.

Waste disposal based on containment in pit disposal systems with eventual chemical and biological degradation by natural processes is a workable concept.

RECOMMENDATIONS

The cost of chemical analyses of waste disposal systems can be greatly reduced if only liquid samples are collected, extracted and analyzed for pesticide residues and degradation products. These results can be used as valid indicators of the safe operation of an economical disposal system if it is designed to minimize leakage and to allow for repair of cracks and ruptures. Such designs are feasible for most all geographical areas.

More effort should be devoted to the development of analytical procedures for measuring the products of the decay of synthetic organic chemicals. These procedures would be useful for the analyses of the contents of disposal systems and the environment in general.

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Detailed interpretation of the analytical data and the result of our experience in the sampling and analyses of over 70 different disposal systems

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could be used to make a variety of recommendations about the construction and operation of waste disposal systems. Most of these would be minor and therefore they are not itemized here. Others which are more important have already been presented in this and other sections of this current report.

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SECTION 5

BACTERIOLOGICAL STUDIES

INTRODUCTION

It has been proposed that degradation of pesticides in pesticide disposal pits may offer an attractive method for the disposal of unwanted pesticides. Few disposal pits exist, and no information concerning types and numbers of bacteria inhabiting such systems was available. An understanding of the types and numbers of bacteria present in such a system contributes valuable baseline data necessary for evaluating the overall effectiveness of pesticide disposal by the pit method.

Through a detailed examination of the microbiological parameters of the pesticide disposal pit in operation at the Iowa State University Horticulture Station, it is believed that bacteria play a major role in disposal of pesticides by the pit method.

Sampling Procedures

The macropit at the Horticultural Research Station was used. Four sample pipes were constructed by drilling holes in 7.08-cm diam plastic pipes. These were equipped with 3-mm (inside diam) plastic tubing to allow sampling at various levels. The pipes were placed 2 m apart along the central axis of the pit and were designated A, B, C, and D. Pipe A was placed in the shallow end of the pit and pipe D was placed in the deep end of the pit. Pipes B and C were spaced between pipes A and D. Male luer-slips (Nuclepore Corp., Pleasanton, CA.) were placed on the ends of the plastic tubing to accommodate Luer-Lok syringes.

Aqueous samples that were processed aerobically were collected from the plastic sample tubing with 30-cc Luer-Lok syringes (Beckton, Dickinson and Co., Rutherford, NJ). Twenty-five-ml samples were withdrawn from each tube and placed in sterile prescription bottles.

Aqueous samples that were processed anaerobically were collected with 50-cc Luer-Lok syringes containing O₂-free CO₂. The gas was rendered O₂-free by passage over a heated copper catalyst (Holdeman and Moore, 1973). All anaerobic samples were collected 15.0 cm from the bottom of the pit, placed in 2-liter, round-bottom flasks containing O₂-free CO₂ and processed within 30 min after collection.

Soil samples were collected near each sample pipe after the top layer

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of rocks had been removed.

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Submerged rocks were collected randomly from the surface of the disposal pit and placed in sterile collection jars. Each rock was scrubbed aseptically in sterile, deionized water containing 0.1% (w/v) peptone. Appropriate dilutions of peptone water were plated to determine viable cell numbers. The average rock surface area was about 100 cm².

Plating and Identification Procedures

The types and numbers of organisms from rocks, soil and aqueous samples processed aerobically were determined after samples were plated on Trypticase Soy Agar (TSA, Baltimore Biological Laboratory, Beckton, Dickinson and Co., Cockeysville, MD) and peptonized milk agar (PMA), (Larkin, 1972). Deionized water containing 0.1% (w/v) peptone was used for all dilutions. Plates were inoculated by the spread plate technique with 0.1 ml of each sample or diluted samples. Colonies were counted after the plates were incubated at 30°C for 24 hours.

To determine types and numbers of organisms in the anaerobically processed samples, pre-reduced media and dilution blanks were prepared according to methods described by Holdeman and Moore (1973). Three pre-reduced media were employed: TSA, TSA supplemented with 40% rumen fluid, and TSA prepared with pesticide pit liquid rather than deionized H₂O. Roll tubes (25 X 142 mm, Bellco Glass Inc., Vineland, NJ), containing 10.0 ml of molten (45°C) medium were inoculated, rolled in cool (10-15°C) H₂O, and incubated at 30°C. Colonies were counted after 48 hours of incubation.

After counting, colonies were either picked at random to determine predominant types of bacteria or were selected on the basis of morphological differences to determine minority populations. Pure cultures from aerobically collected samples were grown at 30°C on TSA slants in screw-capped tubes and stored at 4°C. Cell morphology and motility were determined on cultures grown on Trypticase Soy Broth (Difco) for 24 hours at 30°C. All Gram-positive organisms were rod-shaped and formed endospores. These were identified according to Gordon et al. (1973). All Gram-negative bacteria obtained from aerobic samples were identified according to Grant and Holt (1977) and Bergey's Manual of Determinative Bacteriology (Buchanan and Gibbons (ed.), (1974)). Bacteria from anaerobic samples were initially streaked on TSA to verify oxygen tolerance. Since all of these organisms were oxygen-tolerant and Gram-negative, they were identified according to Bergey's Manual, Ewing (1973) and Ewing and Fife (1972).

RESULTS AND DISCUSSION

Differences in viable cell numbers existed between samples collected from different pipes (Table 45). The numbers of viable cells in samples collected at different depths from each sample pipe, however, were much the same.

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TABLE 45. A COMPARISON OF VIABLE COUNTS FROM LIQUID SAMPLES
COLLECTED AT DIFFERENT DEPTHS FROM THE TOP OF EACH
SAMPLE PIPE.

Sample Source, Depth	Average viable cells per ml*
Pipe A, 15 cm	2.7×10^4
Pipe A, 46 cm	1.7×10^4
Pipe A, 76 cm	1.7×10^4
Pipe B, 15 cm	1.6×10^5
Pipe B, 46 cm	7.0×10^4
Pipe B, 76 cm	1.2×10^5
Pipe C, 15 cm	2.4×10^6
Pipe C, 46 cm	1.3×10^6
Pipe C, 76 cm	2.6×10^6
Pipe D, 15 cm	4.4×10^6
Pipe D, 46 cm	1.7×10^6
Pipe D, 76 cm	1.0×10^6
Pipe D, 106 cm	2.4×10^6

* All counts were made on TSA plates incubated at 30°C for 24 hours.

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Log₁₀ viable cells per ml of aerobic, liquid samples are presented in Figure 102. Representative data are presented on counts made only by using TSA because bacterial numbers recovered on TSA and PMA were similar. The mean viable counts per ml collected 46.0 cm from the top of sample pipes A, B, C and D over the entire sampling period were 1.6×10^5 , 2.2×10^6 , 2.5×10^6 and 4.2×10^5 respectively. The highest numbers of viable organisms collected per ml from pipes A, B, C and D over the sample period were 2.6×10^6 , 2.2×10^7 , 1.4×10^7 and 3.1×10^6 , respectively. The lowest numbers of viable organisms collected per ml from these same sample pipes were, respectively, 1.4×10^3 , 2.3×10^4 , 1.5×10^4 and 1.2×10^4 . The pit was used routinely and the sporadic introduction of pesticides most likely contributed to the fluctuation of viable cell numbers.

Numbers of bacteria recovered from pipes B and C (near the middle of the pit) were similar. Numbers recovered from pipes A and D generally were lower than numbers observed at pipes B and C, probably because most pesticide disposals were made at central portions of the pit between pipes B and C and several pesticides and carriers can serve as growth substrates for bacteria (Dagley, 1975 and Matsumura, 1974).

Molds (Fungi Imperfecti) and yeasts were occasionally encountered during the enumeration of plates. These organisms were present, however, at less than 1.0×10^3 colony forming units per ml.

Of 119 randomly selected isolates from pit liquid, 89% were Gram-negative rods; 65% of these organisms were pseudomonads. Several pseudomonads did not produce fluorescent pigments and did not accumulate poly- β -hydroxybutyrate; these bacteria resembled Pseudomonas stutzeri. One pseudomonad was identified as Pseudomonas putida. Twenty-nine percent of the isolates were unidentifiable Gram-negative, motile rods; most were oxidase-negative, facultative anaerobes and probably belonged to the family Enterobacteriaceae. Gram-positive bacteria were represented by the genera Arthrobacter (7%), Micrococcus (3%), and Streptomyces (2%). In another experiment, 20 isolates were selected on the basis of colonial morphology. The results were similar to those reported above, except that representatives of the genera Alcaligenes (2 isolates), Arthrobacter, Mycobacterium and Xanthomonas (1 isolate each) were also identified. The metabolic capabilities of these 20 isolates were determined by examining growth in filter-sterilized pesticide pit liquid. After one loopful of actively growing cells was placed in a tube of sterile pit liquid, the cultures were shaken at 30°C for two weeks. During this period, 6 of the 20 organisms grew well, 13 grew sparsely on sterile pesticide pit liquid and only 1 culture, the Streptomyces sp., was incapable of growing on sterile pesticide liquid.

Viable cells per gram of soil recovered on TSA ranged from 4.0×10^5 to 6.3×10^5 . The types of bacteria recovered from the soil layer are presented in Table 46. Bacillus cereus var. mycoides, B. circulans, B. coagulans, B. globisporus, B. larvae, B. licheniformis, B. macerans, B. polymyxa, and 3 Bacillus spp. that we could not identify were observed. Of the pseudomonads isolated from soil, 4 were Pseudomonas putida and 3 closely resembled

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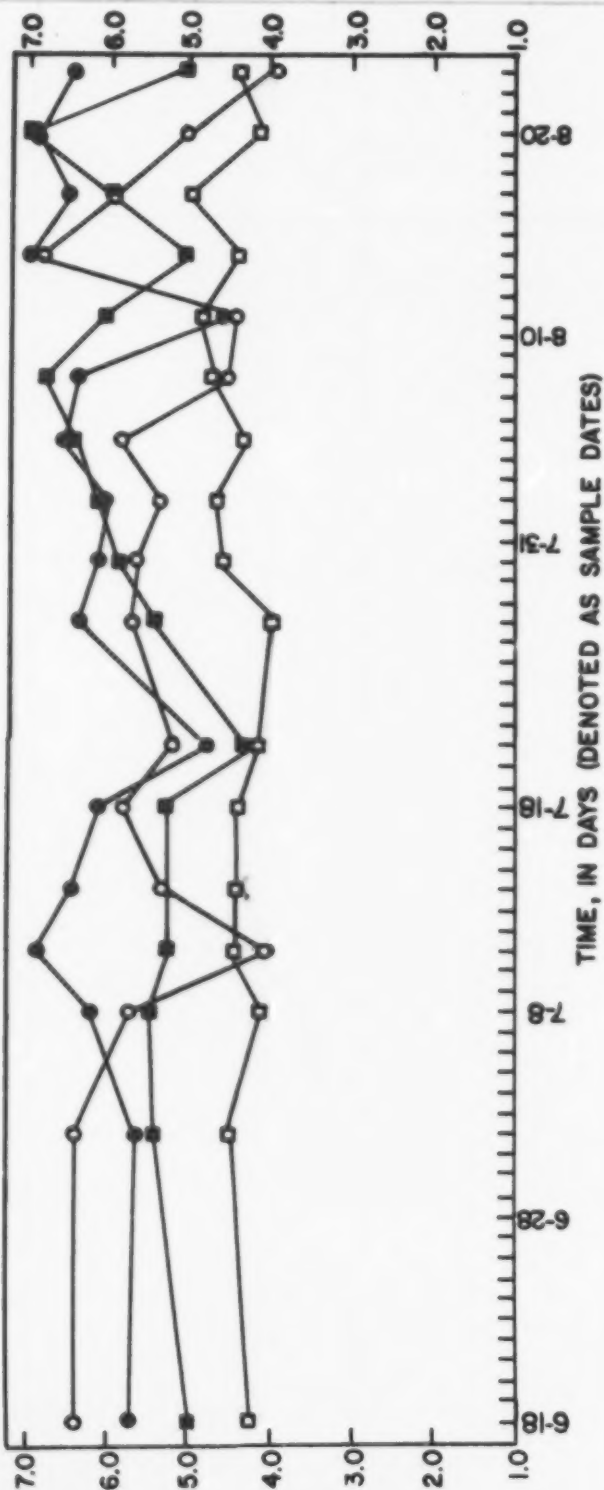


Figure 102. Log 10 viable cells per ml of liquid samples collected 46 cm from the top of the pesticide disposal pit. Samples from four pipes, including pipes A (open squares), B (closed squares), C (closed circles) and D (open circles), were plated on TSA and incubated at 30°C for 24 h. Only data obtained in samples collected from June 18 through Aug. 23 are presented.

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TABLE 46. DISTRIBUTION OF 34 BACTERIA ISOLATED FROM SOIL
SAMPLES AND 40 BACTERIA ISOLATED FROM ROCK SURFACES.

Bacterial group	Number (percent) of isolates from soil*		Number (percent) of isolates from rock surfaces*		Percent of all soil and rock isolates*
<u>Bacillus</u> spp.	17	(50)	8	(20)	33.7
<u>Pseudomonas</u> spp.	7	(21)	17	(43)	32.4
Gram-negative rods	7	(21)	12	(30)	25.6
<u>Citrobacter</u> spp.	2	(4)	0	(0)	2.7
<u>Enterobacter agglomerans</u>	0	(0)	2	(5)	2.7
<u>Arthrobacter</u> spp.	1	(1)	1	(2)	2.7

* Colonies were randomly picked from samples plated on TSA and PMA and incubated at 30°C for 24 hours.

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Pseudomonas stutzeri. Two Citrobacter species were encountered: C. freundii and C. diversus. A larger proportion of facultative anaerobes inhabited the soil than the liquid in the pit.

Types of bacteria isolated from rock surfaces (Table 46) were similar to those isolated from soil, but were different from those isolated from pit liquid. Several members of the genus Bacillus were observed. Four of the 8 Bacillus sp. could not be identified, but 4 were B. coagulans, B. macerans, B. megaterium and B. polymyxa.

Although 1.0×10^4 bacteria were recovered per ml of anaerobically processed pit liquid samples, no strict anaerobes were observed. Colonies were selected from roll tubes on the basis of morphology, and all organisms belonged to the family Enterobacteriaceae. Fourteen of 16 isolates were identified as either Escherichia coli that did not produce gas from glucose at 45°C or Enterobacter agglomerans. The 2 remaining cultures were Citrobacter diversus. Although the genera Vibrio and Aeromonas commonly are isolated from soil or water, none of the bacteria that we isolated from anaerobically processed samples were oxidase positive. Small wildlife typically find shelter under the cover of the disposal pit during winter months. Fecal material from these animals could have served as a source of bacteria belonging to the family Enterobacteriaceae.

The average pH values of samples obtained from each pipe were 7.4 (pipe A), 7.7 (pipes B and D), and 7.8 (pipe C). The highest pH value recorded during the entire sampling period was 8.2, whereas the lowest pH value observed was 6.6.

Minor, day-to-day fluctuations were observed in the temperature of the pit liquid. When the rainfall-activated cover responded to precipitation, the pit was covered and usually cooled 1-2°C if the pit remained covered for 24 hours or more. A difference of 1°C occurred between the shallow and deep ends of the disposal pit. The range of temperatures observed from June to September was 17°C to 24°C; the mean temperature during this period was 20.1°C. During the winter, the liquid in the pit was frozen solid.

The quantitative importance of our data would be more significant if data from a pesticide-free control pit were presented. No control pit was available for study, but data from soil and lake water have been presented by other authors. Edwards (1975) reported that 5.4×10^4 aerobic, heterotrophic bacteria were collected per ml lake water. Jones (1971) obtained counts of 1.9×10^3 viable bacteria per ml of low-nutrient lake water and 8.2×10^3 per ml of high-nutrient lake water during a period of May to November. He also found that bacterial numbers were consistently three orders of magnitude higher in mud (4.2×10^6 per ml) than in water samples. Gram-negative bacteria predominated in lake water samples, and the diversity of genera isolated were significantly greater than the diversity of organisms that we isolated from the pesticide disposal pit. It is difficult to estimate numbers of viable bacteria in a heterogenous ecosystem such as the soil, but 10^8 to 10^{10} viable bacteria per gm of dry soil are commonly reported (Alexander, 1977).

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Although the numbers of bacteria that we isolated from the disposal pit may be lower than numbers isolated from dry soil, the numbers of bacteria isolated from the liquid in the disposal pit were significantly higher than those typically found in lake water. High numbers of bacteria in the liquid portion of the pit may be a result of the wide variety of pesticidal compounds that were introduced into the pit. This influx of pesticides, as well as variations in temperature and pH, could affect microbial populations.

The mere presence of bacteria in the disposal pit does not prove that the bacteria degraded pesticides. Some forms, such as the Bacillus spp. may be dormant, or the bacteria might be adventitious residents whose presence is not associated with the addition of pesticides to the pit. However, most of the isolates that we tested grew in filter-sterilized pit liquid, and it is possible that they did play a significant role in pesticide degradation. Our data indicate substantial biological activity as evidenced by the numbers and the fluctuation of numbers of bacteria in the pit. In addition, gas chromatographic analyses performed on extracts of the pit liquid and soil indicated that there has been no long-term accumulation of any pesticide (G. Junk and J. J. Richard, Iowa State University, personal communication).

Several aspects of pesticide disposal by the pit method are in need of evaluation. An understanding of the effects of the presence and types of soil and (or) rocks is necessary. Our data show that the inclusion of soil and rocks probably resulted in the presence of a more diverse flora than if the pit contained only liquid. It also may be worthwhile to analyze the effects of temperature, pH and the addition of organisms capable of transforming pesticidal compounds. Also, the addition of analogs of pesticides that are readily metabolized may facilitate pesticide degradation.

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SECTION 6

ENTOMOLOGICAL STUDIES

INTRODUCTION

Concern with pesticidal chemicals in the environment has centered around their harmful effects in humans, birds, and other animals. Iowa State University has pit containment systems that are used to dispose of pesticidal chemicals on their research farms. Containment facilities are superior to dumping or other types of land and water disposal because they keep the chemicals from producing unwanted effects in the environment. Since containment for an indefinite period of time is very costly, soil was placed in the pits with the hope of introducing factors that would decompose the pesticides to less harmful compounds. If the pit contents lose their biological activity they can be released into the environment without harmful effects. Chemical analysis techniques are excellent for detecting and measuring levels of specific chemicals in pesticide pits. For that reason the Entomology group developed chemical analysis techniques for parathion, carbaryl, carbofuran, and atrazine. The problem with chemical techniques is that compounds with biological activity may be present but pass undetected. Even if such compounds are detected, their toxicity properties may not be known. For that reason the Entomology group also ran bioassays with extracts of the contents of several pits to determine if the toxicity was similar to levels that would be predicted by chemical analysis.

A bioassay is a test to determine the toxicity of a sample from its reaction with living matter. The major limitation of bioassays is that all organisms respond differently to chemicals. This means that even bioassays can not eliminate all concerns over the toxicity of pit contents. With increased numbers and diversity of species that are used for bioassay tests, there is increased probability of detecting compounds with adverse biological effect. In this study, only insects were used to bioassay pit samples because (1) insects are easier to rear than other animals, (2) insects can be handled in large numbers, and (3) insects are very sensitive to insecticides, a class of pesticides that produce harmful effects in most animals.

METHODS

Sampling

Agronomy and Agricultural Engineering Minipits--

Insecticides were added to minipits at the Agronomy and Agricultural Engineering Research Center (AAERC) according to the plan shown in Table 47.

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TABLE 47. INSECTICIDE ADDITIONS TO MINIPITS AT THE AGRONOMY AND AGRICULTURAL
ENGINEERING RESEARCH CENTER, IOWA STATE UNIVERSITY ON APRIL 20, 1977

Insecticide and formulation	Insecticide additions to minipits			Minipit experimental conditions†
	Rate	Actual insecticide* (by weight)	Insecticide formulation (g)	
carbaryl (Sevin®)‡ 50% wettable powder	1 x	0.5	300	with nutrients aeration nonaeration
	0.05 x	0.025	15	without nutrients aeration nonaeration
parathion§ 15% wettable powder	1 x	0.5	300	with nutrients aeration nonaeration
	0.05 x	0.025	15	without nutrients aeration nonaeration
carbaryl + parathion (+ four herbicides)	1 x carbaryl + 1 x parathion	0.5	300	with nutrients aeration nonaeration
	0.05 x carbaryl + 0.05 x parathion	0.025	15	without nutrients aeration nonaeration
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TABLE 47. (continued).

* Based on 60 l of water per minipit.

† There were eight minipits per insecticide and eight pesticide combination minipits (containing two insecticides and four herbicides) for a total of 24 minipits containing insecticides.

‡ The carbaryl formulation (EPA Reg No. 1016-41; EPA Est 635-OH-1, 7021404) and analytical and technical grades of carbaryl were provided gratuitously by Dr. D. E. Thurman, Union Carbide Corporation (Agricultural Products Division, 909 Blanco Circle, P.O. Box 1906, Salinas, CA 93901).

§ The parathion formulation (EPA Reg No. 769-77; EPA Est 769-CA-01) and analytical and technical grades of parathion were provided gratuitously by J. M. Magner, Monsanto (Monsanto Agricultural Products Company, 800 N. Lindbergh Boulevard, St. Louis, MO 63166). The 15% parathion wettable powder came from Woolfolk Chemical Works, Inc. Box 938, Fort Valley, GA 31030) and the analytical and technical grade samples came from a Monsanto manufacturing plant at Anniston, AL.

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There were a total of 56 minipits; 16 of the minipits contained insecticides, 32 contained herbicides, and 8 contained a mixture of two insecticides and four herbicides. A detailed description of the minipits is included in Section 3, Description of Systems Evaluated. Addition of pesticides to these minipits occurred April 20, 1977. Analytical samples were drawn from the thoroughly mixed contents of the minipits at 0, 1, 2, 4, 8, 12, 16, 20, 24, 52, and 68 weeks following addition of the pesticides.

Soil plus water samples were taken in 118 ml glass jars while stirring the contents of the minipits. To correct for evaporation (in all but the 0-week samples), water was added to each minipit just before sampling to restore the volume to 60 l. The water added to insecticide-containing minipits is shown in Table 48. Samples were held at 2°C until they were analyzed. Water and soil portions of each sample were well separated at the time the samples were extracted. Characteristics of these samples are shown in Table 49.

TABLE 48. CUMULATIVE AMOUNTS OF WATER (LITERS) THAT WERE ADDED TO E, F, AND G SERIES MINIPITS

Time after insecticide addition	Series	E (carbaryl)	F (parathion)	G (carbaryl, parathion + 4 herbicides)
28 weeks	Mean	9.34	10.27	11.02
	Range	7.51-10.56	9.48-11.78	7.49-13.7
52 weeks	Mean	12.92*	12.21†	13.87
	Range	11.58-15.19*	9.71-13.68†	7.87-16.53
68 weeks	Mean	16.93*	15.95†	18.67
	Range	15.01-18.75*	12.74-17.77†	11.06-22.19

* Results are based on 5 pits because of excessive water loss in 3 pits that fractured during the winter.

† Results are based on 7 pits because of excessive water loss in 1 pit that fractured during the winter.

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TABLE 49. CHARACTERISTICS OF SAMPLES FROM MINIPITS IN THE E, F, AND G SERIES THAT WERE USED FOR CARBARYL AND PARATHION ANALYSES (ALL DATA ARE THROUGH WEEK 68 AND REPRESENT 96 SAMPLES FOR EACH SAMPLE CHARACTERISTIC IN EACH SERIES)

Sample Characteristics	Series	E (carbaryl)	F (parathion)	G (carbaryl, parathion + 4 herbicides)
Dry weight of soil (g)	Mean	24.46	25.67	25.42*
	Range	19.79-33.70	19.56-35.67	19.65-34.56
Water removed from samples (ml)	Mean	82.1	80.7	76.6
	Range	71.0-95.0	66.0-92.0	63.0-90.5
pH	Mean	7.1	7.0	6.8
	Range	6.3-7.9	6.3-7.8	6.1-7.5

* All dry weights of soil for week 1 were lost; soil data are for 88 samples only.

Horticultural Research Station Macropit and Micropits--

Water and soil samples were collected for bioassay from eight sites in the Horticultural Research Station macropit and from the four micropits (Figure 103). A detailed description of these pits is found elsewhere in this report. The surface rock layer was removed from each site in the macropit. One liter of water was removed from above the soil surface at each site. The eight water samples were placed in separate glass jars. At each site, two or three cores were removed from the top four to six inches of soil using an auger. The eight soil samples (100 to 200 g per sample) were placed in separate glass jars. The rock layers were lifted off each of the micropits. Water was sampled in the same way as from the macropit. Four small "handfuls" of surface soil were removed from each micropit. The four "handfuls" came from different quarters of the pit, and were combined into one sample. The four soil samples (100 to 200 g per sample) from the micropits were placed in separate glass jars.

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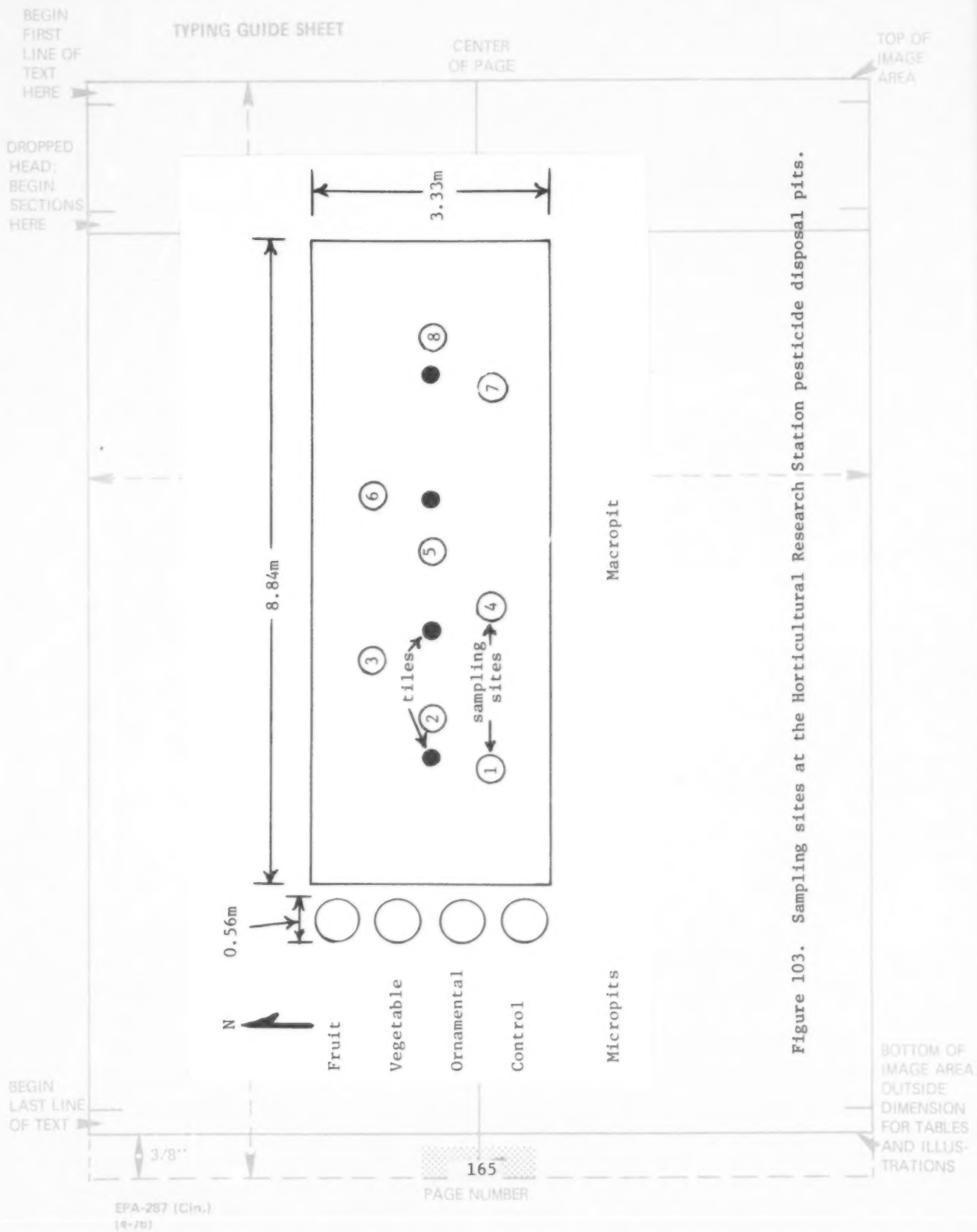


Figure 103. Sampling sites at the Horticultural Research Station pesticide disposal pits.

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Chemical Analysis of Soil and Water Samples

Parathion and Carbaryl--

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Extraction--Brief summaries of extraction and analytical procedures used for minipit samples from the AAERC follow. The water layer was pipetted into a graduated cylinder and extracted in a separatory funnel with 75, 50, and 50 ml of diethyl ether. The combined diethyl ether extracts were filtered through anhydrous Na_2SO_4 and the filtrate reduced to 50 ml under partial vacuum (Buchler/Searle flash evaporator, model PTFE-1GN). The wet soil and glass jar were weighed and the soil extracted with 75, 50 and 50 ml of an acetone:Benzene:methanol (1:2:1 by vol.) mixture. The jars were shaken (Fisher-Kahn shaker, ≈ 280 oscillations/min and ~ 32 mm stroke distance) for one hour for each extraction. The jars were centrifuged for 15 minutes ($\sim 600 \times g$) to precipitate the soil and the supernatant liquid was decanted. The combined liquid from the three extractions was reduced to about 25 ml under partial vacuum. The liquid was extracted in a separatory funnel with three 50-ml portions of diethyl ether. The combined diethyl ether extracts were filtered through anhydrous Na_2SO_4 and the filtrate reduced to 50 ml under partial vacuum.

Recovery experiments were performed using 56 mg of the same formulation of 50% wettable powder carbaryl that was added to the minipits (see Table 47, footnote $\dagger$), 28 g soil (wet weight, containing 8.23% moisture) similar to that added to the minipits, and 70 ml of tap water. The carbaryl concentration in relation to the wet weight of the soil was 1,000 ppm. Using the extraction procedures described above and triplicate samples, an average recovery of 96.5% was obtained from the carbaryl, soil, and water mixture. Recovery experiments using 100 mg of the same formulation of 15% wettable powder parathion added to the minipits (see Table 47, footnote $\S$), 28 g soil (wet weight, containing $\sim 9.62\%$ moisture) similar to that added to the minipits, and 60 ml of tap water showed that these extraction procedures removed essentially 100% of the added parathion from the water and soil portions.

When parathion and carbaryl were present in the same sample, the final part of the extraction procedure was modified. After reducing solvent extracts to dryness with partial vacuum (Buchler/Searle flash evaporator, model PTFE-1GN) and mild heat (40°C) the residue was dissolved in 50 ml of diethyl ether. Half of this (25 ml) was transferred to a sample jar for parathion analysis by gas chromatography. The remaining solution (25 ml) was evaporated to dryness with partial vacuum and mild heat. The residue was dissolved in 25 ml of methanol for colorimetric analysis of carbaryl.

Carbaryl derivatives--Carbaryl must be derivatized before it can be analyzed by gas-liquid chromatography (GLC) because of its thermal instability. The electron capture detector (ECD) is very sensitive to the N-tri-fluoroacetyl derivative of carbaryl. Some of the metabolites of carbaryl can also be detected by this derivatization procedure. Derivatization was done according to Seiber (1972). Sample cleanup was necessary prior to the derivatization. Carbaryl was also derivatized using heptafluorobutyryl (HFB)

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anhydride, and the HFB-carbaryl analyzed by GLC. Derivatization of carbaryl was done in ethyl acetate at 100°C according to Seiber (1972). Dilutions for GLC were prepared in hexane. This procedure was not used on samples because a colorimetric procedure described later by McDermott and DuVall (1970) was faster and quite sensitive for the comparatively large quantities of carbaryl and 1-naphthol present in the extracts.

Gas-liquid chromatography--GLC was used for the analysis of parathion and paraoxon. Appropriate volumes (usually 5 μ l) of the water and soil extracts (or dilutions thereof) were injected into a Varian, gas chromatograph (model 3740) equipped with a ^{63}Ni , electron capture detector. The column was about 2 mm (i.d.) x 91 cm packed with 3% Apiezon[®] N on Chromosorb[®] G (60-80 mesh). Operating temperatures were: injector 200°, column 180°, and detector 300°C. Nitrogen flow was about 38 ml/min. Technical parathion (99.4% pure); paraoxon, diethyl 4-nitrophenyl phosphate (99% pure); and aminoparathion, 0,0-diethyl 0-(4-aminophenyl) phosphorothioate were used as reference standards. Parathion and aminoparathion were received gratuitously from Monsanto Agricultural Products Co., St. Louis, MO and Bayer Pflanzenschutz, Leverkusen, West Germany, respectively.

Analysis of carbaryl derivatives was done on a 4 mm (i.d.) x 200 cm glass column packed with 3% DC-200 on 80/100 mesh Gas Chrom[®] Q. Operational conditions were: injector temperature 220°C, column temperature 200°C, ^{63}Ni ECD temperature 300°C, and nitrogen carrier gas 40 ml/min.

Spectrophotometry--The analytical method of McDermott and DuVall (1970) is based on the reaction of 1-naphthol and p-nitrobenzene-diazonium fluoroborate to form a colored compound with an absorbance maximum at 475 nm. In a solution of KOH in methanol, the carbaryl was saponified to 1-naphthol, which was determined colorimetrically. A neutral dilution of the sample extract was carried through the colorimetric procedure and used as a zero point for the measurement of the sample color on the spectrophotometer. The neutral solution also gave an estimate of hydrolyzed carbaryl (1-naphthol) in the sample extracts. This colorimetric method has been shown to be specific for measuring carbaryl content in mixed formulations in admixture with 13 different pesticides.

Appropriate dilutions of water and soil extracts of the minipit samples were carried through the colorimetric procedure. Samples of carbaryl, technical grade (Lot. No. 74215, Assay 100%, Shipment No. 15-ARD-34, Register No. 511-01-0965) and carbaryl, analytical grade (Ref.: 11128-27, Assay 99.9%, Shipment No. 15-ARD-34, Register No. 511-01-0965) were received gratuitously March 2, 1977 from Union Carbide Corporation, Salinas, CA. Dilutions of methanol solutions of both technical and analytical grade samples were used with the colorimetric procedure and gave essentially identical standard curves. The carbaryl, technical grade was used, therefore, as the standard for comparison with extracts of the minipit samples. The colorimetric procedure for carbaryl yields data for both 1-naphthol (neutral solution in the procedure) and 1-naphthol + carbaryl (caustic solution). The carbaryl values are derived by subtracting 1-naphthol values (neutral solution) from 1-

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naphthol + carbaryl values (caustic solution).

Carbofuran and Atrazine--

High-pressure liquid chromatography (HPLC) was used to develop methods for analyzing pesticides in soil and water. Literature reviews of analysis of pesticides by HPLC and extraction of atrazine and carbofuran from soils were made. The latter two chemicals are widely used as an herbicide and as a soil insecticide, respectively.

Extraction and cleanup--Brief summaries of the procedures used for extraction and cleanup of atrazine and carbofuran from soil, adapted from Gorder et al. (1980), follow. Twenty-five grams of soil were weighed into 100-ml sample jars and extracted three times with 50 ml of a methanol:0.1 N HCl (1:1, by volume) mixture. The jars were shaken (Fisher-Kahn, ~280 oscillations/min and ~32 mm stroke distance) for 15 min for each extraction. The jars were centrifuged for 15 min (~600 x g) to precipitate the soil and the supernatant liquid was decanted into a separatory funnel. The combined liquid from the three extractions was partitioned with three 100-ml portions of methylene chloride. The combined methylene chloride extracts were filtered through anhydrous Na₂SO₄ and the filtrate reduced to 5 ml under vacuum on a rotary evaporator at 30°C.

A glass column, 22 mm (i.d.) by 20 cm was packed with successive layers of 10 g anhydrous Na₂SO₄, 11 g Florisil activated for 1 h at 110°C, and 20 g anhydrous Na₂SO₄. The packing was wetted with 25 ml hexane. The 5-ml methylene chloride phase was added to the column and eluted with 150 ml of 35% ethyl acetate in hexane (v/v). The eluate was evaporated to dryness under vacuum on a rotary evaporator at 30°C. The residue was made up to 15 ml with methylene chloride and pushed through a silica Sep-pak® (Waters Associates). The pesticide was eluted from the Sep-pak with 3 ml of methanol. The eluate was measured, transferred to a 7.4-ml sample vial and stored at -45°C until analyzed by HPLC.

Recovery experiments adding 100 µg of atrazine (analytical standard, 99.2% purity from CIBA-GEIGY Corp., Greensboro, NC) and carbofuran (analytical standard, reference MEK 628 from FMC Corp., Middleport, NY) to 50 g of soil produced recoveries of 95.05 ± 9.67% for atrazine and 115.50 ± 10.44% for carbofuran.

High-pressure liquid chromatography--Using a Waters Associates µBondapak C₁₈ column and an absorbance wavelength of 254 nm it is possible to obtain distinct peaks of atrazine and carbofuran with aqueous solutions of acetonitrile (40% v/v) or methanol (60% v/v) as the mobile phases. These solvents provide a good separation of at least 1 min between peaks of atrazine and carbofuran and also retain good sensitivity of about 10 ng for atrazine and 50 ng for carbofuran (Figure 104).

Atrazine, carbofuran and some of their metabolites have been measured using the HPLC system and an aqueous solution of methanol (60% v/v) as the mobile phase with a flow rate of 2 ml/min. The retention times under these conditions are listed in Table 50.

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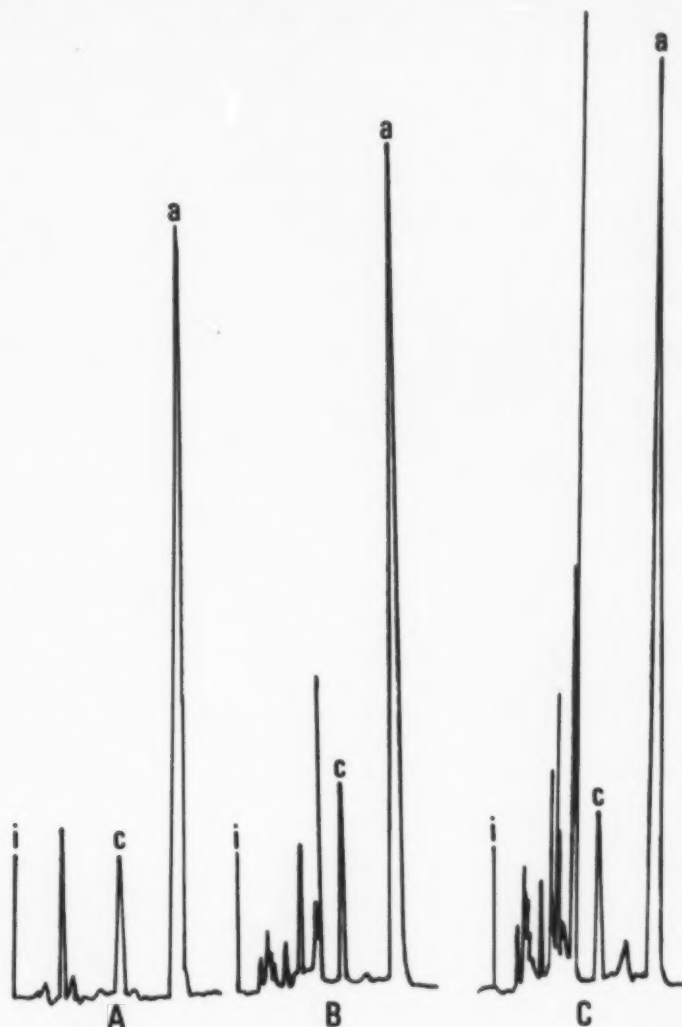


Figure 104. Tracings of sample chromatograms from HPLC using a Waters Associates μ Bondapak C₁₈ Column, U6K injector, Model 6000A Solvent Delivery pump and 440 UV Detector. Mobile phase was 60% methanol in H₂O (v/v). Flow rate was 1 ml/min. Peak a = atrazine; c = carbofuran; i = injection.

- A. Standard solution of 250 ng atrazine and 250 ng carbofuran
- B. Extract of untreated soil (50 g) with 100 μ g of each pesticide added
- C. Extract of soil (25 g) from field treated with atrazine and carbofuran.

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TABLE 50. RELATIVE RETENTION TIMES OF VARIOUS PESTICIDES
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Pesticide	Retention time* (min)
atrazine	4.2
2-Cl, 4-amino 6-ethylamino 1,3,5-triazine	2.1
2-Cl, 4-amino 6-isopropyl amino 1,3,5-triazine	2.3
carbofuran	2.7
3-keto carbofuran	2.3
3-hydroxy carbofuran	1.9

* Obtained at a chart speed of 0.42 cm/min.

Bioassay of Soil and Water Samples

Rearing Procedures--

The test insect, Drosophila melanogaster (Oregon R-C strain), was obtained from the California Institute of Technology (Pasadena, CA). This strain has been widely used for insecticide bioassays. The flies were maintained in a rearing room with a mean temperature of 20°C (range 16.7° to 25.6°C) and a relative humidity range from 45% to 68%. Except during times of colony maintenance, the room was kept completely dark. The flies were reared in wide-mouth, one-quart, glass jars. The standard diet consisted of cornmeal, agar, sugar, yeast, and propionic acid. The adults that emerged in the jars were removed after a 24-hour period. These flies were held in the rearing room for 3 days in jars containing applesauce or standard diet.

Samples and Insecticide Standards--

Initially, Horticultural Research Station pit samples were extracted in Entomology labs using procedures similar to those described for minipit sample extractions. The LD₅₀ values determined from these samples were compared with simultaneously determined LD₅₀ values for standard samples of dieldrin and parathion. Dieldrin was substituted for carbaryl in the bioassay procedures owing to the low toxicity of carbaryl toward the test insects. These bioassays gave poor agreement between bioassay results and gas chromatographic data from Energy and Mineral Resources Research Institute (EMRRI) personnel. For that reason, a third important insecticidal component of the Horticultural Research Station pits, endosulfan (Thiodan®), was used as the bioassay standard. At the same time we stopped extracting our own samples and used the

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same samples that had been analyzed by gas chromatography by EMRRI project personnel. The results showed good agreement between chemical analytical data from EMRRI and our bioassay data.

All insecticide standards (analytical grade) were checked for purity by thin-layer chromatography using both UV light and colorimetric spray reagents. Stock solutions of each insecticide were prepared in acetone and preserved at -4°C .

Bioassay Methods--

The preparation to be tested and the insecticide standard were added to cylindrical, wide-mouth glass jars (approximately 118 ml in volume and 5 cm x 8 cm outside diameter and heights, respectively). In tests of samples collected in 1978 and 1979, untreated soil extract from the control micropit was added to all sample and standard jars so each jar contained the same volume of soil extract. These solvents were evaporated to dryness under a stream of nitrogen. A corn oil solution containing 0.3 ml of corn oil in 100 ml of acetone (0.5 ml/jar in early tests and 0.25 ml/jar in tests of 1978 and 1979 samples) and acetone (1 ml/jar) were added to allow even coating of the jar walls with the soil extract and insecticide standard. Coating was accomplished in a fume hood using a jar roller especially built for this purpose (Figure 105). The corn oil facilitated contact of the insecticides by the test insects. Screw top lids with screen centers were used to cover the jars. Metal caps attached to the lids were used as feeding trays to prevent flies from dying due to starvation and desiccation during the tests. Applesauce was used as food in early tests, but the rearing diet was used in tests of all 1978 and 1979 samples. Approximately 50 flies were then counted into each coated bioassay jar using the methods described by Edwards et al. (1957). One hour was allowed to pass before insects were added to the jars. Addition of insects to control jars (acetone and corn oil) immediately after rolling resulted in 23% mortality; a one-hour delay reduced this death rate to 4.6%.

Each bioassay had two types of controls. The first control was used in which the flies had only food; neither solvent nor corn oil was added to that jar. High mortality in this control would indicate that the flies were weak or that the jar or food was contaminated. The second control had the jar walls coated with untreated soil extract, the residue from 1.0 ml acetone, and 0.5 ml corn oil. High mortality in this second control and low mortality in the first control (food only) would indicate contaminated pipettes, untreated soil extracts, acetone, or corn oil solution; or addition of flies before the acetone had totally evaporated.

The bioassay jars were held in the rearing room and kept in darkness by means of a cloth cover for 24 hours. At the end of this period, dead flies in each jar were counted. Moribund flies showing movement but unable to move from one area of the jar to the other were counted as dead. The live flies were killed using chloroform and the total number of flies for each jar was counted.

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When known quantities of endosulfan were used to fortify pit extracts, one of three extracts tested masked the expected toxicity of the added endosulfan.

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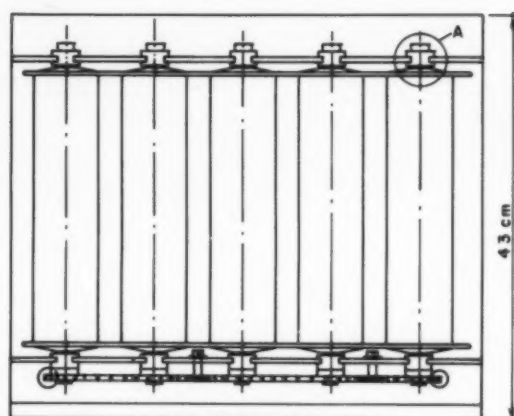
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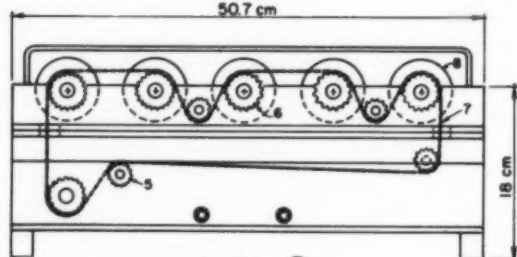
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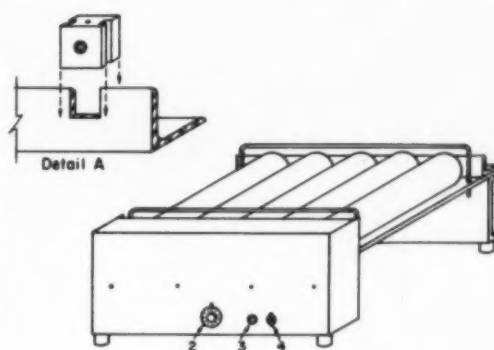


Top View (1)

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Front View (2)



End View (3)

Figure 105. Variable speed jar roller for insecticide film bioassays.
1 = electric motor, 2 = variable speed rheostat, 3 = fuse,
4 = switch, 5 = idler sprocket, 6 = drive sprocket on
rubber roller, 7 = chain drive, and 8 = rubber roller.

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This masking effect could not be corrected by adding extract from the control micropit to the endosulfan standards; it was possibly due to antagonistic action of other chemicals in the pit. The source of antagonism was not obvious from gas chromatographic data reported by EMRRI. The antagonism could be due to a wetting agent or other non-pesticidal component in the extract. Less masking occurred in the affected sample when the extracts were unfortified than when they were fortified. For that reason, only unfortified bioassays were conducted on pit extracts. The unfortified bioassays require substantially more extract than fortified bioassays, so only extracts containing substantial quantities of insecticide were tested.

Data were recorded on coded bioassay data sheets. All information was transferred to computer cards. A computer program similar to the single line program described by Daum (1969, 1970) was used to estimate LD₅₀ values. This program is based on the method of maximum likelihood presented by Finney (1971). LD₅₀ values were estimated graphically for some samples that gave reasonable results but failed to give LD₅₀ values by computer analysis. This was done by drawing the best line through points obtained by plotting percent mortality (corrected for control mortality) versus dose on logarithmic-probability paper. Samples that gave erratic results (such as fluctuating high-low mortality rates) were reanalyzed using adjusted toxicant concentrations until an LD₅₀ level was established. The LD₅₀ values of test solutions were expressed in terms of grams of soil or water. The LD₅₀ values of standard solutions of parathion, dieldrin, and endosulfan were expressed as micrograms of insecticide/jar. The insecticide standard LD₅₀ value divided by the sample LD₅₀ value gave equivalent ppm of the standard insecticide in the sample. For example, if the extract of 50 g of soil was contained in 25 ml of solvent (acetone), 1 ml of solvent contained the toxicants (test solution) in 2 g of soil. Appropriate dilutions of the solvent solution were used for bioassays. Assuming that the LD₅₀ value for the test solution was equivalent to 1 g of soil and the LD₅₀ value for the parathion standard on that day was 1.00 µg of parathion/jar, the soil sample would contain insecticidal toxicants equivalent to 1 ppm of parathion.

RESULTS

Chemical Analyses

Results of chemical analyses for 1-naphthol, carbaryl, and parathion are discussed and shown graphically in the section on chemical studies. Amino-parathion at concentrations similar to parathion concentrations used for preparing standard curves (e.g., 4 to 10 ng/µl) does not give any chromatographic response. In only one or two instances was a small chromatographic peak observed at a retention time similar to that for paraoxon. It seems that paraoxon is not a metabolic product of parathion under these experimental conditions or, if it is, that paraoxon is hydrolyzed to diethyl phosphoric acid and p-nitrophenol or reduced to aminoparaoxon (diethyl 4-aminophenyl phosphate). Diethyl phosphoric acid, p-nitrophenol, aminoparaoxon, and aminoparaoxon are of negligible toxicity compared with parathion and paraoxon.

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Bioassay Analyses

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These bioassays quantitated the response of fruit flies to pit extracts but by themselves provided no clue regarding the toxic chemical(s) causing the mortality. Initial bioassays were run using parathion and dieldrin as insecticide standards. Results obtained from these bioassays did not establish any quantitative relationship between the toxicity of the sample and the amount of any insecticide in the pit samples. Later bioassays were run using endosulfan as the insecticide standard. Results from these bioassays showed that toxicity of the pit samples followed very closely the amount of endosulfan present in the sample. For that reason, the results suggest that increased sample toxicity to fruit flies was mainly owing to increased levels of endosulfan. This was probably also true in earlier bioassays that used parathion and dieldrin as insecticide standards.

Bioassays of samples collected at the Horticulture macropit during 1977 were tested against parathion and dieldrin standards. Because those insecticides were not important components of the samples the equivalent ppm of these compounds in the samples have little meaning. The numbers, however, do serve to indicate how toxic a sample was relative to another sample. In 1977, samples were collected at 8 sites in the macropit (Figure 103) and bioassays were run on samples from all of these locations. The relative toxicities of the soil extracts show a very uneven distribution of toxicants (Table 51). In most cases, water samples contained much smaller amounts of toxicants. The highest amounts of insecticidal materials in the macropit were collected at sites 1 and 4 near the front of the pit. Most of the toxic material was adsorbed to soil, probably explaining why there was little movement of toxic materials to other locations in the macropit. There was an accumulation of insecticidal materials at sites 1 and 4 over the period of sampling owing to additions of insecticides to the pit at these two locations. There was not much increase of insecticidal materials over the sampling period at other sites in the macropit.

TABLE 51. THE TOXICITY OF HORTICULTURAL RESEARCH STATION DISPOSAL PIT SAMPLES COLLECTED DURING 1977 AS DETERMINED BY BIOASSAYS WITH DROSOPHILA MELANOGASTER

Sample site*	Equivalent ppm of parathion†			Equivalent ppm of dieldrin†		
	March 1	June 6	August 2	March 1	June 6	August 2
1	0.70	6.10	12.45	1.11	5.93	12.90
2	<0.76	3.21	5.75	<0.88	3.12	5.95
3	<0.78	2.03	1.27	<0.91	1.98	1.32
4	7.39	21.50	65.13	9.88	20.90	67.49

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TABLE 51. (Continued)

Sample site*	Equivalent ppm of parathion†			Equivalent pp of dieldrin†		
	March 1	June 6	August 2	March 1	June 6	August 2
5	4.81	1.50	1.15	6.43	1.46	1.20
6	<0.71	0.62	0.44	<0.91	0.60	0.45
7	0.57	<0.97	0.80	0.76	<0.94	0.83
8	1.49	2.34	1.13	1.74	2.27	1.21

* Sample sites correspond to those in Figure 103.

† Toxicity is expressed in equivalent ppm of parathion and dieldrin. Equivalent ppm were obtained by dividing the ng of parathion and dieldrin at the LD₅₀ by the mg of sample at the LD₅₀.

These results show that insecticidal materials were segregated in the pit. It was difficult by these procedures to determine if the mean level of insecticides in the pit was increasing or decreasing. For that reason, samples from the 8 sites were composited in 1978 and 1979, and analyses were run on the composited samples.

Soil extracts from the Horticultural Research Station macropit and the vegetable micropit were tested against endosulfan standards during 1978 and 1979. The results are shown in Figures 106 and 107. The bioassay results were very similar to the GLC results for endosulfan as reported elsewhere in this report. This suggests that endosulfan was the actual toxic component in the extracts and confirms the GLC results for endosulfan. Data from the macropit samples showed increased toxicity over the two-year period (Figure 106). This indicates that endosulfan is being added to the pit faster than it is breaking down. The trend in the vegetable micropit (Figure 107) is not as pronounced.

Samples from the control, ornamental and fruit micropits at the Horticultural Research Station were not tested because these pits received little or no insecticides. Soil extracts from the control and ornamental micropits were used successfully as untreated soil extracts in bioassays of the other extracts. Chemical analytical data showed little insecticidal material in the water extracts so these were not bioassayed during 1978 or 1979.

Continued monitoring of the Horticultural Research Station pits with bioassays would probably continue to confirm endosulfan levels found by GLC. Bioassay data would be of concern if these values exceeded those obtained by GLC for endosulfan. An explanation could be provided by running bioassays

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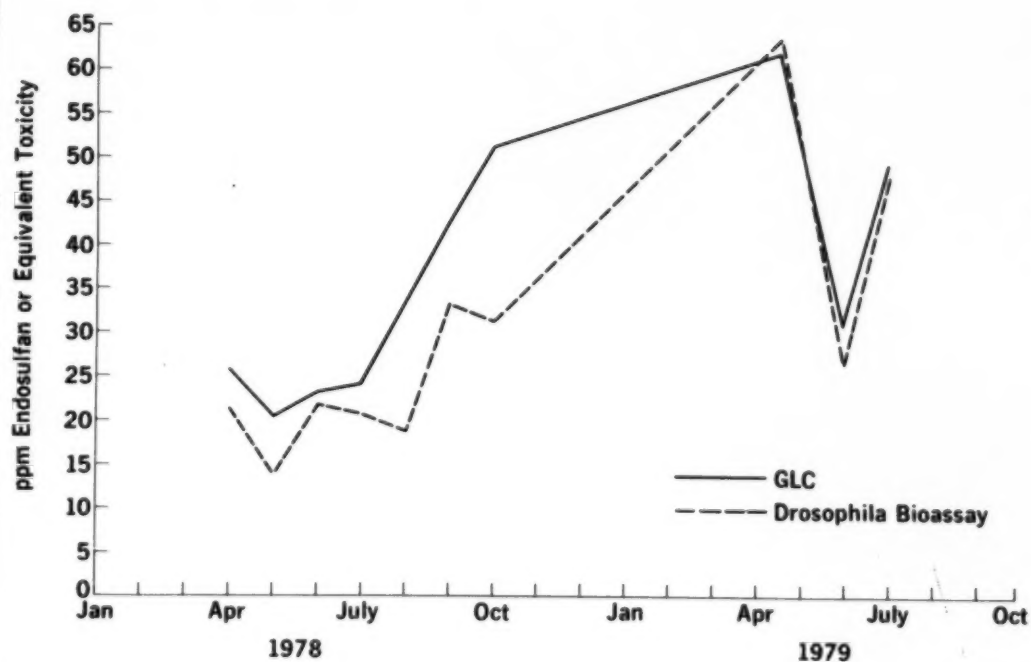


Figure 106. Endosulfan levels in Horticultural Research Station macropit soil extracts, 1978-79.

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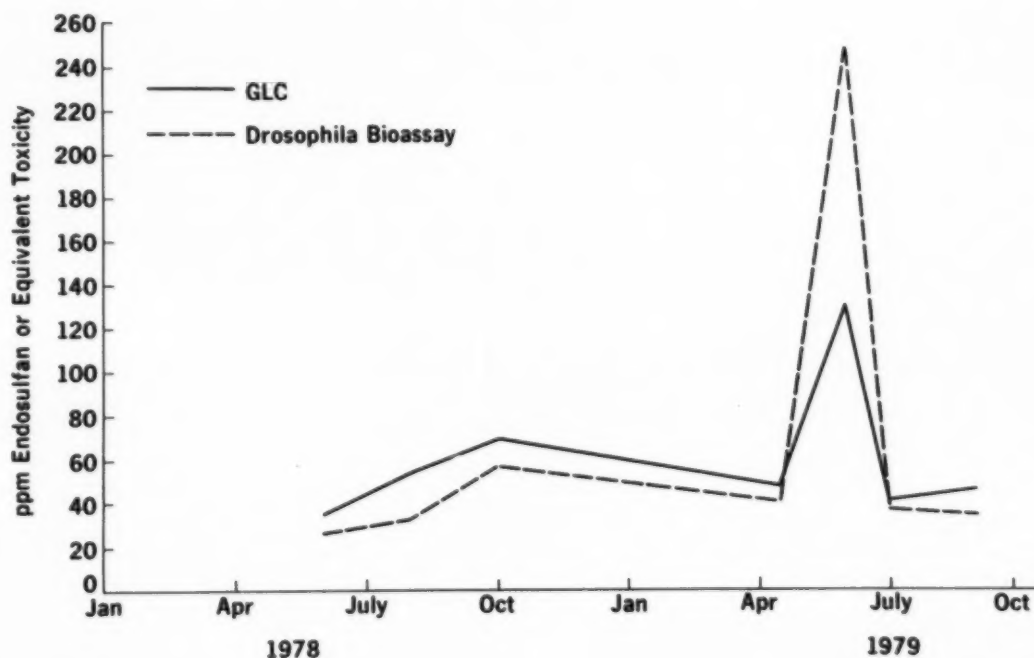


Figure 107. Endosulfan levels in vegetable micropit soil extracts, 1978-79.

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using suspected new chemicals or chemical metabolites as standards. Chemical assays would also have to be performed to confirm the presence of the correct quantity of the new toxicant. The testing of samples for biological toxicity is very time consuming and the results are not specific for any chemical. Bioassays may, however, allow the detection of toxic components that would not be shown by chemical analyses.

Insecticidal bioassays lack specificity, therefore chemical assays are usually better for quantitative analyses. In addition bioassay procedures are quite time consuming. A single film bioassay, as performed in these experiments, consisted of 30 jars and required about 12 hours. This includes maintenance of Drosophila melanogaster colonies, conducting the bioassays, and recording and computing the data. Even though bioassays provide valuable data, they should not be used to replace specific chemical analyses. Bioassays can be used to confirm chemical analyses of insecticides and indicate toxic insecticide metabolites. The probability of detecting specific classes of insecticides by bioassays improves with the use of diverse test species.

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SECTION 7

ESTIMATING PESTICIDE VOLATILIZATION

INTRODUCTION

The approach taken in estimating the magnitude of the role of volatilization in pesticide dissipation in disposal pits was to attempt to relate pesticide volatilization to water evaporation. Although it is possible to measure pesticide volatilization directly, the rate, and the amount, of pesticide volatilization from a pit depends on many environmental (temperature, wind speed) and physical (pit size; pit construction, i.e. walls, cover, filling material, if any; depth to liquid surface) factors. Hence, in order to predict volatilization for different pits in different climates, many direct, involved measurements would have to be made for the numerous possible combinations of conditions. However, the rate and amount of evaporation of water is very easily measured (and to some degree, predictable) and the same factors that affect pesticide volatilization affect water evaporation. Therefore water evaporation integrates all these factors and can serve as an indirect method of estimating pesticide volatilization. There are two differences between pesticide and water vaporization; one is the background levels in ambient air which affects the concentration gradient between air at the surface of the liquid and the air some distance remote from the liquid surface. For water the background concentration is significant relative to that at the surface of the liquid and directly related to the relative humidity (i.e., at 50% R.H., the background level would be one-half that at the liquid surface); for pesticides, the background concentration is very low relative to that at the liquid surface and for all practical purposes can be assumed to be zero (for very huge pits or under conditions of recent pesticide application nearby, this may be slightly in error).

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The second difference relates to the heat required for vaporization. The rate of vaporization of water from a liquid surface is determined in part by the diffusion of water vapor from the stagnant air immediately above the liquid surface into the surrounding air. If the surrounding air is moving at some velocity greater than zero, the water vapor is being removed as it is formed at a rate increasing with wind velocity. Of course if the wind velocity is very great, as in a wet bulb thermometer measurement of relative humidity, the rate of vaporization becomes limited, not by molecular diffusion, but by the rate of transfer of heat to the liquid surface to provide the latent heat of vaporization for water. For pesticides of interest, the rate of volatilization is enough lower so that heat transfer is not limiting. For pits, smaller than lakes, and in contact with soil and rocks, it is assumed that heat transfer by conduction is sufficient and that water evaporation upwind does not significantly affect the background relative humidity of the ambient air.

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If diffusion then controls pesticide and water vaporization from a liquid surface, the rates of diffusion according to Ficks first law are:

$$Q_w = -D_w \frac{\partial C_w}{\partial X} \quad 7.1$$

$$Q_p = -D_p \frac{\partial C_p}{\partial X} \quad 7.2$$

where subscripts w and p refer to water and pesticide, respectively, and Q is the diffusion rate (gm/cm²-day), $\partial C/\partial X$ is the concentration gradient ($\frac{\text{gm/cm}^3}{\text{cm}}$), and D is the proportionality constant between diffusion rate and concentration gradient, called the diffusion coefficient (cm²/day). D is a positive number; the negative sign takes into account water or pesticide movement in the direction of decreasing concentrations.

If we assume that at some distance L from the liquid surface, background levels are reached (zero for pesticides, the ambient relative humidity for water), equations 7.1 and 7.2 become

$$Q_w = D_w \frac{(1-R.H./100)}{L} (VP_w M_w/RT) \quad 7.3$$

$$Q_p = D_p \frac{(VP_p M_p/RT)}{L} \quad 7.4$$

where VP is the saturation vapor pressure of water or pesticide at the temperature under consideration, M is the molecular weight, R is the gas constant and T is the absolute temperature.

Dividing equation 7.4 by 7.3 yields

$$\frac{Q_p}{Q_w} = \frac{D_p}{D_w} \frac{VP_p M_p}{VP_w M_w (1-R.H./100)} \quad 7.5$$

From kinetic theory the average molecular velocity is proportional to $(1/M)^{1/2}$; therefore, the diffusion coefficient should be proportional to $1/M^{1/2}$ or

$$D_w = K/M_w^{1/2} \quad 7.6$$

$$D_p = K/M_p^{1/2} \quad 7.7$$

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where K is the proportionality constant (same for both water and pesticides).
Substituting into equation 7.5 yields

$$\frac{Q_p}{Q_w} = \frac{VP_p M_p^{1/2}}{VP_w M_w^{1/2}} (1-R.H./100) \quad 7.8$$

Equation 7.8 gives the ratio of the rate of pesticide volatilization to water evaporation from a saturated water solution of a pesticide. For less than saturated solutions, VP_p will be less than the saturated vapor pressure as given by Henry's law for dilute solutions or

$$VP'_p = VP_p C'/C_0 \quad 7.9$$

where VP'_p is the vapor pressure of a pesticide with solubility C_0 at equilibrium in air over a solution of concentration C' . Hence for a solution at some concentration C' , less than the saturation concentration C_0 , equation 7.8 becomes

$$\frac{Q_p}{Q_w} = \frac{VP_p M_p^{1/2} C'/C_0}{VP_w M_w^{1/2}} (1-R.H./100) \quad 7.10$$

Table 52 presents the results of calculations using equation 7.8 for saturated solutions of selected pesticides. The ratio of pesticide volatilization to water evaporation is expressed in ppm (however, in this case defined as parts of pesticide volatilized per million parts of water evaporated; of course the pesticide and water in the gas phase would no longer be associated). It is evident from this table that the saturated vapor pressure is very important in determining the relative rates of volatilization of pesticides to evaporation of water. Temperature and the ambient relative humidity are important, but less so. Increasing temperature increases the ratio of pesticide volatilization to water evaporation because ΔH_{vap} for the three herbicides listed are higher than for water, and hence vapor pressures of these pesticides increase with temperature faster than for water. Increasing ambient relative humidity also increases the ratio as less water is evaporated at higher relative humidities, everything else being equal. Vernolate and trifluralin were the only pesticides listed where the ratio of pesticide to water evaporated would exceed the concentration in a saturated solution. In these cases, diffusion of pesticide within the liquid phase to the pesticide-depleted surface layer could limit volatilization and reduce the true values to less than the numbers shown.

The laboratory and field work associated with this phase of the project involved an attempt to verify that the derived relationships between pesticide volatilization and water evaporation were valid, and to gather water evaporation data from the macropit at the AAERC to use in estimating the magnitude of pesticide volatilization.

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TABLE 52. CONCENTRATIONS OF SELECTED PESTICIDES IN EVAPORATED WATER FOR DIFFERENT TEMPERATURES AND RELATIVE HUMIDITIES AS PREDICTED FROM EQUATION 7.8

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Pesticide Properties*	Temp °C	V.P.† mmHg	Relative Humidity				
			0%	25%	50%	75%	87.5%
					ppm		
Vernolate (mole. wt., 203.4; solubility, 90 ppm @20°C)	15 25 35 40	4.08×10^{-3} 1.04×10^{-2} 2.04×10^{-2} 3.79×10^{-2}	1070 1470 1630 2300	1430 1960 2170 3070	2140 2940 3250 4600	4290 5880 6500 9210	8580 11770 13000 18410
Propachlor (mole. wt., 211.7; solubility, 580 ppm @20°C)	15 25 35 40	1.07×10^{-4} 2.30×10^{-4} 4.69×10^{-4} 6.58×10^{-4}	28.7 33.2 38.1 40.8	38.2 44.3 50.8 54.4	57.4 66.4 76.2 81.5	114.7 132.8 152.2 163.1	229.5 265.5 305.0 326.2
Trifluralin (mole. wt. 335.3; solu- bility, <1ppm @27°C)	15 25 35 40	3.01×10^{-5} 1.13×10^{-4} 3.89×10^{-4} 7.01×10^{-4}	10.2 20.5 39.8 54.7	13.5 27.4 53.1 72.9	20.3 41.0 79.6 109.3	40.6 92.1 159.2 218.7	81.2 164.2 318.3 437.3
Parathion (mole. wt., 291.3; solubility, 20-25 ppm @20°C)	15 25 35 40	2.25×10^{-5} 6.25×10^{-5} 1.63×10^{-4} 2.57×10^{-4}	7.1 10.6 15.5 18.7	9.4 14.1 20.7 24.9	14.1 21.2 31.1 37.4	28.3 42.3 62.2 74.7	56.6 84.6 124.3 149.4
Alachlor (mole. wt., 269.8; solubility, 242 ppm @25°C)	15 25 35 40	6.8×10^{-6} 2.2×10^{-5} 6.6×10^{-5} 1.9×10^{-4}	2.1 3.6 6.0 13.0	2.7 4.8 8.1 17.7	4.1 7.2 12.1 26.6	8.2 14.3 24.2 53.2	16.5 28.7 48.4 106.3
Atrazine (mole. wt., 215.7; solubility, 33 ppm @27°C)	15 25 35 40	1.3×10^{-7} 6.5×10^{-7} 2.9×10^{-6} 1.1×10^{-5}	0.03 0.09 0.24 0.69	0.05 0.13 0.32 0.92	0.07 0.19 0.48 1.38	0.14 0.38 0.95 2.75	0.28 0.75 1.90 5.50
Cyanazine (mole. wt., 240.7; solubility, 171 ppm @25°C)	15 25 35 40	5.9×10^{-10} 4.1×10^{-9} 2.5×10^{-8} 1.3×10^{-7}	0.0002 0.0006 0.0022 0.0086	0.0002 0.0008 0.0029 0.0115	0.0003 0.0013 0.0043 0.0172	0.0007 0.0025 0.0087 0.0344	0.0013 0.0050 0.0173 0.0687

* From Weed Science Society of America (1974) and Canadian Department of Agriculture (1968).

† Calculated from the Gibbs-Helmholtz equation for temperatures for which no data existed.

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Laboratory

Laboratory studies of pesticide volatilization were made using volatilization chambers. The physical variables considered important were temperature, relative humidity of influent air, airflow rate, and method of introducing air. Measurements were made at 25 and 40°C; 0, 50, and 100% relative humidities; airflow rates in the range of 5-1000 ml/min; and airflow over (non-aerated) and through (aerated) the pesticide mixture.

The first set of two volatilization chambers used were stainless steel cylinders, 79.6 cm long, 16.3 cm in diameter, with the axis in a horizontal position with an inlet on one end to receive air flow and an outlet on the opposite end with a ground glass connection to the traps. The inlet end caps could be completely removed to provide access to the chambers. For the non-aerated case, the air inlet position was above the liquid level; for the aerated case, the inlet was connected to a gas dispersion tube which was positioned close to the bottom of the cylinder.

The air used was from the central university supply line, pressure regulated to 10 KPa. A manifold was set up to supply four air lines; two for each of the two volatilization chambers. Air from one of the two lines for each chamber was passed through drying tubes filled with drierite to remove the water present (R.H. = 0%); air from the other line for each chamber was bubbled, by means of fritted glass gas dispersion tubes, through a series of two flasks filled with distilled water (maintained at the desired temperature by placement in the water bath) to provide water-saturated air (R.H. = 100% at the temperature of interest). Air flow rates through the four lines were regulated (at ambient temperature) by four flow controllers. Thus total flow through a chamber was regulated by the sum of the two flows, and the relative humidity of the influent air was controlled by the relative amounts of the dry and water-saturated air.

The temperature of a 150.0 insulated water bath containing the volatilization chambers was maintained at either 25±.1°C or 40±.1°C with a 1000 watt constant temperature circulator. Two cold traps in series were used to condense the water and pesticide vapors from the outflow air. The cold traps were maintained at -78°C with dry ice in dewars. Glass beads within the cold trap flasks were used to increase the surface area available on which condensation could occur. The assemblies are shown in Figure 108.

In checking the trapping efficiency of the cold traps, it was determined that essentially all of the water vapor was trapped by the first cold trap. For pesticides, the first trap was at least 50 times more efficient (on a total mass trapped basis) than the second; therefore, it was concluded that two traps would be sufficient. One problem that was encountered was plugging and reduced air flow from the formation of ice in the first trap. By decreasing run times, and thus the amount of water condensed, and changing the apparatus and procedure slightly (switching the inlet and exit), this problem was

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eliminated. Another problem arose in the aerated case, where foaming of the pesticide-water mixture occurred to the extent that foam came through the outlet. A defoaming agent, Agri 2 (TM), was used to correct this problem. The preliminary runs were used in determining run times for sufficient (with respect to analysis) pesticide to volatilize, and in determining experimental procedures for extraction and analyses of test pesticides.

The first volatilization chambers were used in two configurations. One was to fill each chamber about $3/5$ full with 10 liters of the water-pesticide mixture; this resulted in a liquid surface of 1275 cm^2 in each chamber and 6.6 liters of air space over the liquid. This configuration was used to make measurements under saturated (with respect to both pesticides and water) conditions. In the other configuration, used to make measurements under unsaturated conditions, either one or three 8.5 cm I.D. cylindrical glass dishes of pesticide-water mixture were used, resulting in either 57 or 170 cm^2 of liquid surface from which volatilization could occur.

Later in the project, a second volatilization assembly was constructed which could be used in a balance enclosure which allowed an accurate and continuous monitoring of water vaporization, although the assembly was subject to ambient temperature and relative humidity conditions. It was also constructed so that all surfaces in contact with pesticide vapors could be extracted. It was comprised of a 8.0 cm O.D. cylindrical glass dish (7.5 cm I.D.) used as the pesticide-water mixture containers with a second 8.5 cm I.D. cylindrical glass dish inverted over the first. The first dish was set on a balance pan, the second held in place with clamps, did not touch the first. It also had a ground glass outlet in the top through which air could be drawn by an air pump (beyond the cold traps). The assembly is shown in Figure 109.

The procedure used for either assembly consisted of timed runs long enough for a measurable amount (1-10 g) of water and pesticide (a few μg) to be trapped in pre-weighed cold traps. After thawing of the collected water, the traps were reweighed to determine the exact amount of water collected. Benzene was used to extract and dissolve the pesticides trapped; roughly 40 ml was used for the first cold trap and 10 ml used for the second. This benzene was first used to wash the surfaces exposed to pesticide vapors, other than the cold traps, in the second assembly. After extraction by shaking, the benzene was decanted from the water and glass beads and a portion used for pesticide analysis. A Tracor 220 Microtek gas chromatograph with a N163 electron capture detector was used to quantify the pesticides dissolved in the benzene. Sample peak heights were compared to peak heights of a series of standard solutions (concentrations of successive standards increasing by the factor of the square root of 2). Dilutions of samples and variations in the volumes injected were used to obtain amounts (1 ng range) that were optimum for analyses.

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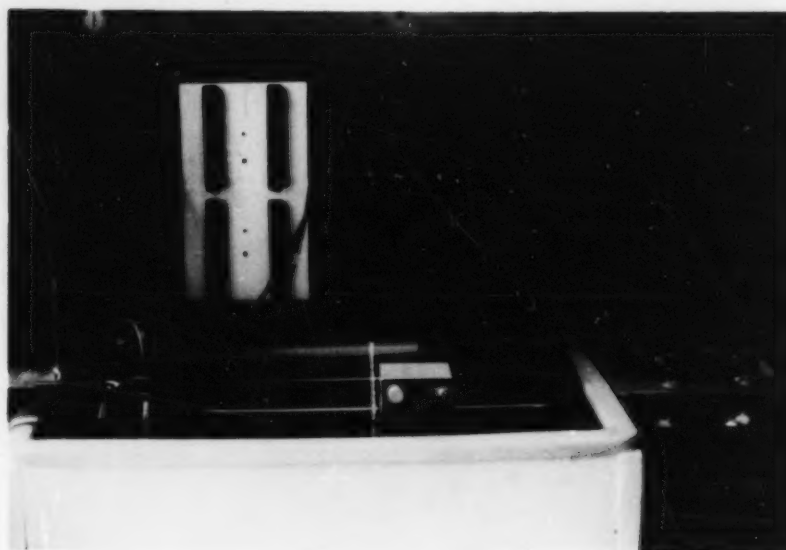


Figure 108. Volatilization study apparatus (stainless steel tanks).

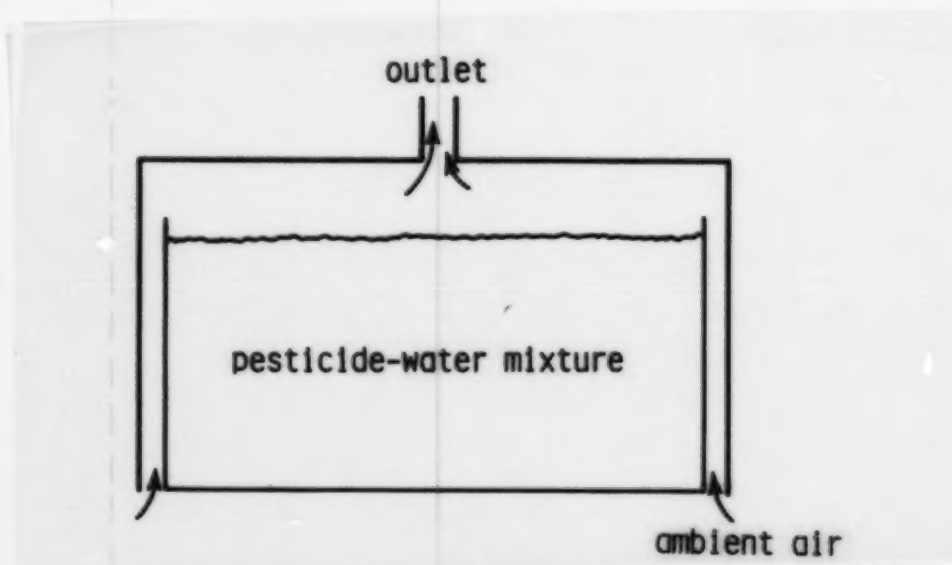


Figure 109. Volatilization study apparatus (glass flasks).

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To estimate pesticide volatilization from water evaporation, depth to the water surface was monitored at the AAERC macropit. This was done using a point gauge and the access to the water surface provided by the five sampling tiles (described in Section 3). Depth readings were taken about weekly and were made relative to the rock surface. In March 1979, a water stage-recorder (FW-1) was installed to provide continuous depth reading with time between manual readings.

In order to relate depth readings to volume measurements an equation was derived for the total pit volume versus depth. The area in m^2 of the water surface in the AAERC macropit is given by:

$$A = 113.5 - 89.0 D + 16.0 D^2 \quad 7.11$$

where D is the depth to the water surface from the rock surface of the pit. The area decreases with depth because of the 2:1 sloping walls of the pit. At the bottom of the pit (D = 1.676 m), the area is $9.3 m^2$. Therefore, the volume of the pit below any depth, D, is given by the equation:

$$V = \int_0^D A \, dD = 90.32 - 113.5 D + 44.5 D^2 - 5.33 D^3 \quad 7.12$$

1.676

A plot of volume-depth is shown in Figure 110. Also shown in the figure is the water storage volume of the pit as a function of depth assuming the porosity of the 0.457 m (18") surface layer of rock was 50% and the drainable porosity of the soil below was 4.2% (as measured by the addition of 676 liters of water 11/22/78).

Precipitation, temperature (air and soil), relative humidity, wind, and pan evaporation data were taken and reported by AAERC personnel.

Problems encountered in 1978 with the measurements at the macropit involved unexplained decreases in the depth to the liquid surface. There are at least three sources for this problem: one, rainfall blew in; two, unrecorded additions were made to the pit; and three, the plastic pit liner has a hole(s) in it. From the continuous depth-time readings obtained with the stage recorder in 1979 it is apparent the first two sources are real. To check the extent of a leak in the plastic liner, if any, soil samples to about 3 m were taken at the corners of the pit (and one between the old and new pits, and one remote from both pits). These soil (and water) samples were analyzed for pesticides and the results are reported in the section on chemical studies. For the most part the levels found were very low relative to the pit contents. Another indication that if a leak was occurring it was not massive is the fact that the depth to the soil water surface at the time the samples were taken (7/18/79) was about 1080 mm greater than the depth to the liquid surface within the pit.

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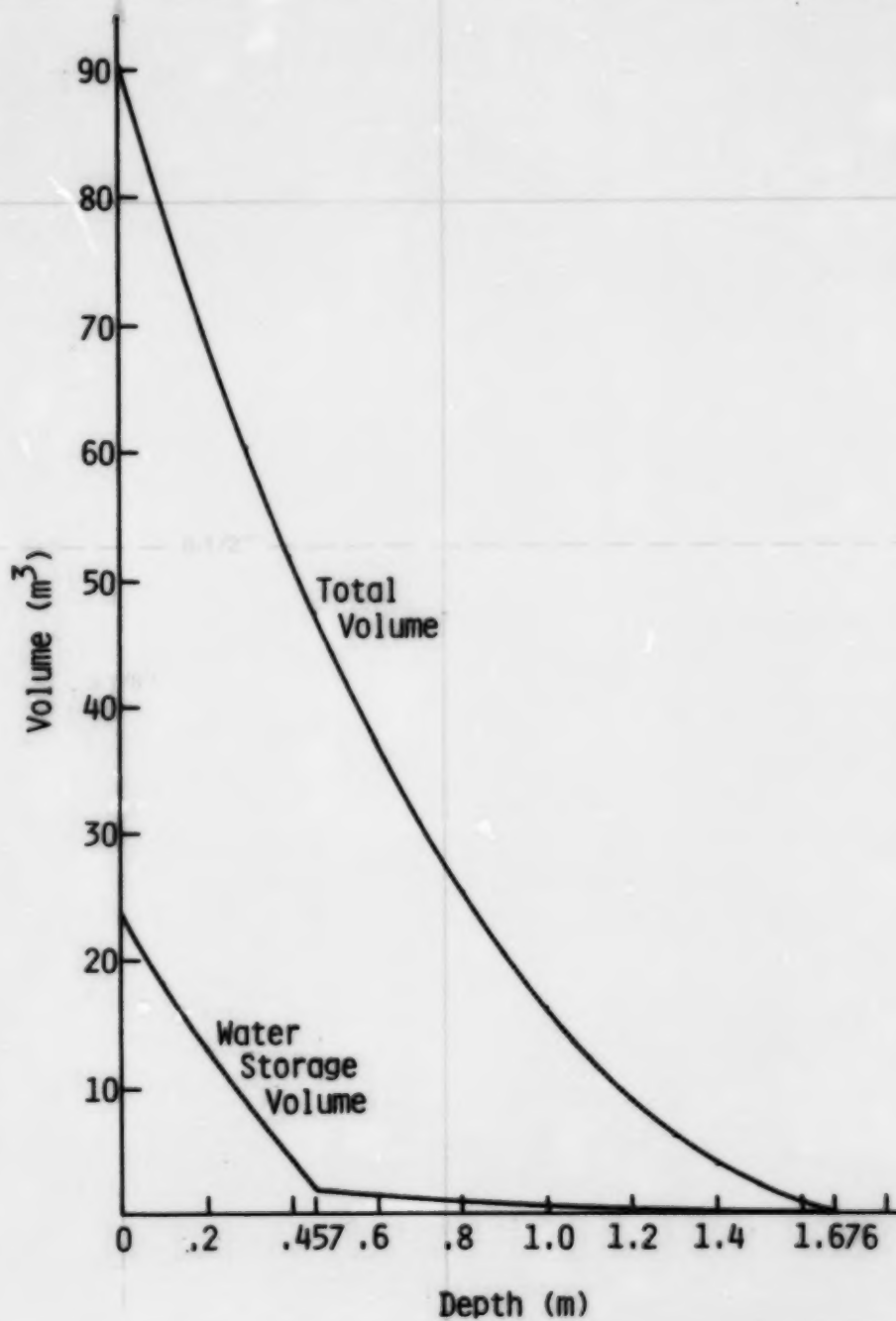


Figure 110. Volume-depth relationships, AAERC macropit.

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Measurements of atrazine, alachlor, and propachlor volatilization from aqueous solutions under saturated vapor conditions were made. Table 53 lists the data for alachlor, propachlor, and water-vapor concentrations in exiting air from the volatilization chambers for the various conditions of inflow-air relative humidity, air-flow rate, and temperature studied. The chambers were more than half full and had 1275 cm² of surface from which volatilization could take place. Each value listed is an average of at least three runs. Values for atrazine were zero; no atrazine vapor was detected in exiting air. The values in parentheses are herbicide vapor concentrations corrected for air-flow rates based on the assumption that exiting air is saturated with water vapor (i.e. the correction factor equals 23.1 mg/l at 25°C, or 51.1 mg/l at 40°C, divided by the measured water vapor concentrations; values for water-vapor concentrations different than 23.1 or 51.1 are believed due to errors in air-flow measurement).

The effect of the relative humidity of the incoming air on the herbicide vapor concentrations is minimal if present at all; in some cases, concentrations are slightly higher at 0% R.H., in other cases at 100% R.H. Aeration of the pesticide-water mixture caused substantial (up to 100%) increases in the alachlor vapor concentrations over the non-aerated case. The fact that these increases are much greater for the high air flow rate indicates that the increases may be due to suspended aerosols. These aerosols would have less time to settle back to the surface of the liquid with the 1 l/min air flow rate relative to the 0.25 l/min rate.

As expected the increased temperature of 40°C relative to 25°C resulted in a substantial (about 300%) increase in herbicide vapor concentrations. Surprisingly there was a slight decrease in the propachlor concentrations at both temperatures and alachlor concentrations at 40°C for the non-aerated cases at the decreased flow rate. (This may be due to partial adsorption to exposed surfaces before the vapor reached the cold trap as discussed later).

Table 54 relates the degree of saturation of herbicide in the vapor phase to that in solution. As discussed earlier, for the fairly dilute solutions under consideration, Henry's law should be valid and it would be expected that the degree of saturation in the vapor phase would equal that in the solution phase. For propachlor the agreement is within experimental error; however, there is a significant difference for alachlor (which could result if the literature value for the vapor pressure of alachlor is in error).

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An attempt was made to determine the ratio of water to herbicides volatilized under conditions where the air was not saturated with either water or herbicide vapor. Table 55 shows the results obtained. This work was done using the same volatilization chambers, air flow rate, temperatures and herbicide concentrations as previously used; however, the liquid surface was much less. In the 1-dish chamber the source of water and herbicide for volatilization was a mixture contained in a cylindrical glass dish 8.5 cm in diameter;

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TABLE 53. HERBICIDE CONCENTRATIONS IN AIR UNDER SATURATED CONDITIONS

R.H. %	Conditions		Alachlor		Propachlor		Water*	
	Air Flow l/min	Temp. °C	aerated	non-aerated ng/l	aerated	non-aerated ng/l	aerated	non-aerated ng/l
0	0.25	25	365(380) [†]	334(309)	533(555)	581(537)	22.2	25.0
50	0.25	25	472(355)	432(302)	686(514)	729(509)	31.0	33.3
100	0.25	25	367(367)	305(281)	528(527)	506(517)	23.1	25.0
0	1.0	25	544(586)	265(289)	722(788)	633(664)	21.3	21.6
50	1.0	25	468(506)	290(309)	534(579)	501(533)	22.4	22.9
100	1.0	25	462(478)	286(294)	555(575)	487(503)	23.1	23.1
0	0.25	40	753(925)	843(948)	1129(1386)	1284(1445)	41.62	45.28
50	0.25	40	750(822)	903(822)	1102(1200)	1385(1257)	47.06	56.44
100	0.25	40	652(730)	631(730)	931(1032)	932(1077)	46.07	44.89
0	1.0	40	1258(1318)	1011(1013)	1526(1596)	1570(1571)	48.81	50.81
50	1.0	40	1175(1206)	835(894)	1641(1686)	1635(1751)	49.33	47.83
100	1.0	40	1372(1441)	1035(1041)	1578(1658)	1699(1708)	48.53	50.57

* At 25°C, saturated water vapor concentration is 23.1 mg/l; at 40°C, 51.1 mg/l (at 1 atmosphere pressure).

† Corrected values in parentheses.

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TABLE 54. DEGREE OF HERBICIDE SATURATION IN AIR AND AQUEOUS SOLUTION PHASES

Alachlor				Propachlor			
Vapor		Solution		Vapor		Solution	
ng/l*	% of sat.	ppm	% of sat.	ng/l*	% of sat.	ppm	% of sat.
Temp = 25°C							
207	93	124	51	544	21	168	29
Temp = 40°C							
908	--†	124	--†	1468	--†	168	--†

* Data are averages of vapor concentrations from non-aerated chambers, with flow rate corrections made on the basis of water trapped (i.e. 100% R.H. of exiting air assumed).

† Data not available to calculate percents of saturation.

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TABLE 55. HERBICIDES VOLATILIZED UNDER UNSATURATED CONDITIONS

Conditions		Alachlor				Propachlor				Water	
		1-dish		3-dish		1-dish		3-dish		1-dish	3-dish
Air- flow Temp. °C	R.H. % /min.	Conc. ng/ℓ	Pest/ Water ppm	Conc. ng/ℓ	Pest/ Water ppm	Conc. ng/ℓ	Pest/ Water ppm	Conc. ng/ℓ	Pest/ Water ppm	Conc. mg/ℓ	Conc. mg/ℓ
25	1	25	3.3	28	2.6	51	6.8	90	8.3	7.5	10.9
25	0	29	3.8	33	2.8	46	6.0	79	6.6	7.7	11.9
25	0	23	3.4	--	--	44	6.5	--	--	6.8	--
25	0	24	3.8	28	2.7	44	7.0	81	7.8	6.3	10.4
25	0	24	3.8	29	2.9	45	7.1	82	8.1	6.3	10.1
25	0	27	4.3	35	3.4	58	9.2	103	9.9	6.3	10.4
25	0	25	3.3	39	3.3	65	8.7	106	8.9	7.5	11.9
25	0	24	3.5	54	4.8	61	9.0	82	7.3	6.8	11.3
25	0	19	2.8	40	4.0	54	8.1	86	8.7	6.7	9.9
25	0	29	3.9	55	5.2	69	9.3	114	10.9	7.4	10.5
25	0	32	4.7	61	5.8	66	9.7	97	9.2	6.8	10.6
25	0	12	1.6	61	5.0	71	9.5	95	7.8	7.5	12.2
Avg.		24	3.5	42	3.9	56	8.1	92	8.5	7.0	10.8
(Std. dev.)		(5)	(0.8)	(13)	(1.2)	(10)	(1.3)	(12)	(1.2)	(10.5)	(0.8)

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Conditions		Alachlor				Propachlor				Water	
		1-dish		3-dish		1-dish		3-dish		1-dish	3-dish
R.H. %	Air-flow l/min.	Temp. °C	Conc. ng/l	Pest/ Water ppm	Conc. ng/l	Conc. ng/l	Pest/ Water ppm	Conc. ng/l	Pest/ Water ppm	mg/l	mg/l
0	1	40	163	11.0	121	4.9	20.0	296	13.4	14.8	24.9
0	1	40	135	9.0	150	5.9	16.5	248	13.4	15.0	25.3
0	1	40	151	10.1	142	5.9	16.9	252	12.4	14.9	24.1
0	1	40	157	10.4	170	7.2	17.9	270	16.5	15.1	23.7
0	1	40	173	12.1	171	7.4	18.6	266	16.1	14.3	23.1
0	1	40	142	10.1	109	4.7	17.1	241	11.9	14.1	23.3
0	1	40	141	9.9	127	5.3	18.3	260	14.6	14.2	23.8
0	1	40	143	9.6	139	5.7	17.0	254	12.1	14.9	24.2
0	1	40	139	9.2	147	5.8	17.4	263	14.5	15.1	25.3
0	1	40	134	8.6	149	6.1	17.7	276	14.0	15.6	24.3
0	1	40	148	10.5	129	5.5	17.7	250	10.6	14.1	23.6
0	1	40	161	10.6	169	6.9	18.0	273	16.3	15.2	24.4
Avg.			149	10.1	144	5.9	17.8	262	13.8	14.8	24.2
(std. dev.)			(12)	(0.9)	(20)	(0.9)	(15)	(40)	(1.9)	(0.5)	(0.7)
100	1	25	47		54			76		22.0	20.6
100	1	25	40		51			67		22.0	22.9
100	1	25	33		48			58		21.7	23.0
100	1	25	36		49			62		23.0	23.0
100	1	25	30		68			69		21.8	24.5
100	1	25	37		58			72		22.5	23.2
100	1	25	33		67			58		23.2	25.1
Avg.			37		54			66		22.3	23.2
(Std. dev.)			(6)		(8)			(7)		(0.6)	(1.4)

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for the 3-dish chamber there were three glass dishes. For the 1-dish chamber, with incoming air at 0% relative humidity, the exiting air at 25 and 40°C averaged 30 and 29% relative humidity, respectively. For the 3-dish chamber the exiting air averaged 47% at both 25 and 40°C. For the 1-dish chamber for propachlor the exiting air averaged 10 and 16% of saturation (based on values for non-aerated case in Table 53) at 25 and 40°C, respectively. For the 3-dish chamber the respective numbers were 16 and 20%. For the 1-dish chamber for alachlor the exiting air averaged 8 and 15% of saturation at 25 and 40°C, respectively. For the 3-dish chamber the respective numbers were 14 and 15%. Based on the percentages given above, it would appear that the ratio of herbicide to water volatilized under unsaturated conditions is about one-third that of saturation. This is close to the ratio of $M_w^{1/2}/M_p^{1/2}$ of 1:3.9 for alachlor and 1:3.4 for propachlor, which takes into account differences of molecular weight on diffusion.

The second volatilization assembly of one cylindrical dish inverted over the other containing the pesticide-water mixture (but subject to ambient conditions) was used to study trifluralin and parathion under unsaturated conditions (with respect to the vapor phase). In general, as would be expected, pesticide and water concentrations in outflow air decreased with increased flow. The pesticide-water mixture was a 0.5% active ingredient suspension and therefore was a saturated solution, with solubilities of trifluralin and parathion being < 1 and ~20-25 ppm, respectively. At 25°C and 50% R.H. the predicted pesticide/water ratio for trifluralin and parathion (from equation 7.8, Table 52) would be 41 and 21 ppm, respectively. The average values in Table 56 are 151 and 7.8 ppm, respectively.

Also given in Table 56 are values for trifluralin and alachlor volatilizing from a sample of the macropit liquid at the AAERC. This sample was saturated with respect to the water solubilities of the two herbicides. Air sampling over the AAERC macropit showed the concentrations of trifluralin and alachlor in air at the rock surface were 0.9 and 7.6 ng/l, respectively. At the time of sampling the liquid surface was about 38 cm below the rock surface of the pit. The difference in the trifluralin-alachlor concentration ratio of 2 for air passing directly over the pit liquid, relative to the ratio of 1/8 for air over the rock-filled pit, may indicate that adsorption to the rock surface has an effect on vapor concentrations and losses.

Field

Tables 57 and 58 present the results of depth to the liquid surface measurements made on the macropit at AAERC. As described in the method section, depths less than 457 mm indicate that the liquid surface is within the rock layer; depths greater than 457 mm indicate that the liquid surface is within the soil layer. During the two years measurements were made, the closest the liquid surface was to the rock surface was on 5/31/79 when the depth was 244 mm. At this point the liquid stored was about 11000 l (pit capacity is about twice this value). For both years during the period of at least May through July the liquid surface was within the rock layer. During this time in 1979 the average evaporation was about 2 mm/day based on the total pit area (equal to 225 m²/day). Then from August through December the liquid surface was with-

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TABLE 56. PESTICIDES VOLATILIZED UNDER SATURATED CONDITIONS

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<u>Conditions</u>			<u>Trifluralin</u>		<u>Parathion</u>		<u>Water</u>		
R.H. %	flow l/min.	Temp. °C	Conc. ng/l	Water ppm	Conc. ng/l	Water ppm	Initial Conc. mg/l	Δ Conc. mg/l	Final R.H. %
55	135	24	1658	240	77	11.2	11.9	6.9	87
52	211	23	1132	193	61	10.3	10.8	5.9	80
44	216	25	1357	124	71	6.5	10.2	10.9	91
44	255	24	1133	110	51	5.0	9.5	10.3	92
54	566	24	535	102	25	4.8	11.7	5.2	78
52	662	24	699	139	45	9.0	11.2	5.0	75

<u>Conditions*</u>			<u>Trifluralin</u>		<u>Alachlor</u>		<u>Water</u>		
36	440	27	137	12	51	4.5	9.2	11.5	81
46	547	24	75	14	45	8.5	9.9	5.3	70

*For pit liquid sample.

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TABLE 57 DEPTH TO LIQUID SURFACE IN MACRO DISPOSAL PIT - AAERC (1978)

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	#1	#2	#3	#4	#5	Avg.	Δ	Additions*
	mm							l
5/31/78 †	319	319	319	319	319	319	--	2578
6/1/78	323	322	322	322	323	322	-3	216
6/5/78	335	335	334	335	334	335	-13	227
6/7/78	327	327	327	327	328	327	8	151
6/9/78	333	334	333	333	333	333	-6	106
6/16/78	315	314	315	315	315	315	18	379
6/23/78	330	329	330	330	329	330	-15	68
6/26/78	312	312	312	312	312	312	18	
6/28/78	324	324	324	324	323	324	-12	
6/30/78	330	330	330	330	330	329	-6	
7/7/78	357	357	358	357	358	357	-27	57
7/14/78	365	366	366	365	365	365	-8	57
7/28/78	401	401	401	401	401	401	-36	114
8/4/78	472	461	465	466	466	466	-65	
8/11/78	---	586	586	587	---	580	-114	57
8/17/78	670	646	664	667	669	663	-83	
8/24/78	559	550	548	544	551	550	113	39
8/25/78	574	561	568	568	573	568	-18	
9/1/78	469	459	473	478	475	470	98	170
9/8/78	576	558	572	546	575	564	-94	
9/15/78	498	490	485	463	490	484	80	
9/22/78	509	498	486	474	473	487	-3	
9/29/78	577	590	614	608	600	597	-110	
10/6/78	821	804	822	821	825	818	-221	
10/13/78	897	896	904	900	895	898	-80	
10/27/78	979	973	975	976	977	976	-78	
11/3/78	991	997	1007	---	---	955	-19	
11/10/78	1042	1041	1040	1037	1040	1040	-45	
11/13/78	748	651	813	800	790	760	280	
11/15/78	804	798	815	---	812	807	-47	
11/19/78	720	707	729	---	726	725	82	
11/22/78	793	785	792	---	790	792	-67	
11/24/79	534	511	536	---	545	532	260	676 §
11/28/78	605	561	607	---	615	597	-65	
12/4/78	750	708	740	746	747	738	-141	
12/8/78	883	876	879	875	876	878	-140	
12/24/78	1040	1030	1033	1030	1032	1033	-155	
Totals							-714	4895

* Additions are of pesticide-water mixtures, do not include unknown volume of rinse water.

† First day depth (from liquid surface) was measured; additions, precipitation and evaporation are for the month of May.

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† No data taken.

§ 676 l of water were added 11/22/78 in a test to estimate drainable soil porosity.

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TABLE 58 DEPTH TO LIQUID SURFACE IN MACRO DISPOSAL PIT-AAERC (1979)

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Date	#1	#2	#3	#4 mm	#5	Avg.	$\Delta \uparrow$	Additions*
1/5/79	1239	1207	1253	----	1239	1235	-202	
1/19/79	1403	1401	1417	----	1407	1407	-172	
2/2/79	1521	1581	1569	1522	1521	1542	-135	
2/16/79	1556	1500	1569	1595	1572	1574	-32	
3/2/79	1528	1587	----	----	1554	1556	18	
3/9/79	1534	1584	----	----	1554	1557	-1	
3/16/79	1537	1584	1599	1576	1566	1572	-15	
3/24/79	381	370	364	376	365	369	1203	
3/31/79	280	279	279	275 §	280	279	90	
4/4/79	288	285	287	----	288	287	-8	
4/11/79	288	288	288	----	289	288	-1	
4/18/79	329	328	328	----	328	328	-40	
4/23/79	326	327	327	----	327	327	-1	
5/2/79	361	362	362	----	361	362	-35	68
5/9/79	327	329	328	----	327	328	34	890
5/16/79	358	359	360	----	358	359	-31	151
5/23/79	312	312	312	----	312	312	47	927
5/31/79	252	241	241	----	240	244	68	749
6/6/79	305	305	306	----	304	305	-61	
6/13/79	326	326	326	----	324	326	-21	
6/20/79	308	311	309	----	308	309	17	
6/27/79	295	297	297	----	295	296	13	38
7/5/79	310	311	311	----	310	311	-15	
7/11/79	337	338	337	----	337	337	-26	
7/18/79	360	360	360	----	359	360	-23	
7/25/79	380	382	381	----	380	381	-21	
8/8/79	422	424	423	----	422	423	-42	
8/15/79	449	449	453	----	448	450	-27	
8/20/79	445	446	443	----	445	445	5	
8/22/79	444	445	443	----	444	444	1	
8/30/79	512	491	498	----	496	499	-55	
9/7/79	530	534	515	----	494	518	-19	
9/14/79	726	666	720	----	612	681	-163	
9/19/79	816	1026	814	----	----	885	-204	
9/26/79	844	839	846	----	737	817	68	
10/7/79	966	857	915	----	809	887	-70	
10/11/79	999	981	990	----	830	950	-63	
10/19/79	1001	1025	1021	----	875	951	-1	
10/26/79	688	665	699	----	726	695	256	
11/2/79	783	728	787	----	741	762	-67	
11/9/79	709	693	622	----	689	678	84	

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Date	#1	#2	#3	#4 mm	#5	Avg.	$\Delta^\dagger$	Additions* x
11/20/79	808	814	820	----	737	795	-117	
11/28/79	732	692	736	----	669	707	88	
12/6/79	791	806	803	----	727	782	-75	
12/10/79	819	834	824	----	744	805	-23	
12/17/79	954	955	954	----	797	915	-110	
Totals							118	2823

* Additions are of pesticide-water mixtures, do not include unknown volume of rinse water.

$\dagger$ Δ is for difference from last reading 12/24/78.

$\ddagger$ Water in vertical tiles finally froze.

§ Stage recorder installed in this tile after depth measurement 3/31/79.

Depth to water table measurements made for area surrounding pit this day.

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in the soil depth; during this time the average evaporation was about 0.4 mm/day. The increase in depth to the liquid surface during the winter months is believed due in part to migration of water up to the shallow colder layers to form frost. The large decrease in depth during the week between 3/16/79 and 3/24/79 is believed due to the melting of this frost and of snow that had drifted into the pit area. On the basis of the average amounts of water evaporated, it is estimated that about 28000 l of water evaporated from the AAERC pit per year.

An attempt was made to correlate evaporation from the macropit with open pan evaporation and environmental factors such as air and soil temperature, wind, relative humidity, etc. In order to do this the data were separated into two groups, that with the liquid surface within the rocks, and that within the soil layer. In addition, data for days on which precipitation occurred (and may have blown into the pit), days on which additions were made to the pit (volume of rinse water not known), and days on which the unexplainable rises in the pit level occurred (suspected due to unrecorded additions to pit) were not included in the analyses. Surprisingly, there was no correlation between water evaporation (in either the rock or soil level data) with open pan evaporation. The pit evaporation during the time the liquid surface was within the soil layer was positively correlated with soil temperature.

SUMMARY

1. From laboratory tests under both saturated and unsaturated vapor conditions, pesticide concentrations in air (ng/l) were not significantly affected by whether or not evaporation of water was occurring (i.e. whether inflow air and 0 or 100% R.H.). Therefore "codistillation" was not believed to be a factor.
2. The increases with temperature of measured pesticide concentrations in air under saturated conditions, and the ratio of pesticide to water concentrations under unsaturated conditions, agreed roughly with those predicted.
3. For both saturated and unsaturated conditions, particularly for propachlor, Henry's law was apparently valid (i.e. the equilibrium vapor pressure over a less-than-saturated solution was equal to the vapor pressure over a saturated solution times the fraction of saturation; errors in solubility or saturated vapor pressure data obtained from the literature could account for some discrepancies).
4. Aeration, by creation of aerosols, may enhance pesticide losses to the air (although initially in the condensed phase and not as a vapor).
5. The use of an equation:

$$\text{Pesticide loss} = \text{water loss} \times \frac{VP_p M_p^{1/2}}{VP_w M_w^{1/2} (1-R.H./100)}$$

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should be valid for estimating pesticide vapor loss from a pit from water loss at a given temperature and relative humidity. This assumes that the pit liquid is saturated with the pesticide of interest; losses would be less for concentrations less than saturations.

Use of the equation above for the AAERC macropit provides an example of the magnitude of the role that volatilization plays in the dissipation of pit-disposed pesticides. In 1978, 24, 7, and 12 kg of alachlor, cyanazine, and propachlor were deposited in the macropit. If roughly 28000 liters of water were evaporated at 25°C and 50% ambient relative humidity, 0.8, 0.0004, and 15% of the deposited alachlor, cyanazine, and propachlor would have been dissipated by volatilization. This shows that for pesticides dumped in significant quantities and with vapor pressures less than or equal to propachlor, volatilization does not play a major role in dissipation (if the pesticides dissipate in one or two years). For the herbicide EPTC, more volatile than vernolate (see Table 52), only one disposal of 2 kg was made in 1978. Under these circumstances, it would be possible to lose all of this deposit due to volatilization.

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SECTION 8

PIT EVAPORATION AS AFFECTED BY CLIMATOLOGICAL CONDITIONS

INTRODUCTION

Deposition of dilute agricultural pesticide wastes into an open, water tight pit appears to be a feasible method of disposal. The volume of continuous disposals into any such container would be limited by the rate of evaporation which in turn is directly related to exposed surface area. Characterization of those factors influencing pit evaporation is essential to determine the surface area required to accommodate a particular volume of continuous disposal. The evaporative characteristics of the pesticide disposal facility macropit located on the Horticultural Research Station at Iowa State University were studied during the 1978 and 1979 growing seasons. Extensive data were collected in an attempt to predict the rate of pit evaporation from climatological and pit related factors. Such a prediction could have utility in matching pit size to disposal needs.

PIT DESCRIPTION

Constructed in 1969-70, the pit has 20.0 cm thick concrete walls and floor and is set in the ground to a depth of about one meter. Its internal dimensions are 8.8 m by 3.3 m with a sloping floor resulting in a depth range of 0.9 to 1.2 m from end to end. The pit is filled with a 25.0 cm thick soil layer sandwiched between two similar layers of coarse gravel. This fill material consumes all the internal depth to within 18.0 cm of the top (Figure 111). A mobile, rain-activated cover operates to limit liquid intake from precipitation. The pit is oriented east-west and is bordered on the East by an equipment and pesticide storage building. This and other nearby buildings and trees interrupt normal air movement across the pit and intercept direct solar radiation during the early morning hours (until 8:00 or 9:00 a.m. during June and July).

The facility has been in operation as a repository for waste agricultural pesticides since its construction. Detailed study of its evaporative characteristics began in 1978 and continued during 1979. Results of this study reflect the evaporative characteristics of this facility under normal usage.

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Climatological data collected on-site included daily measurements of air

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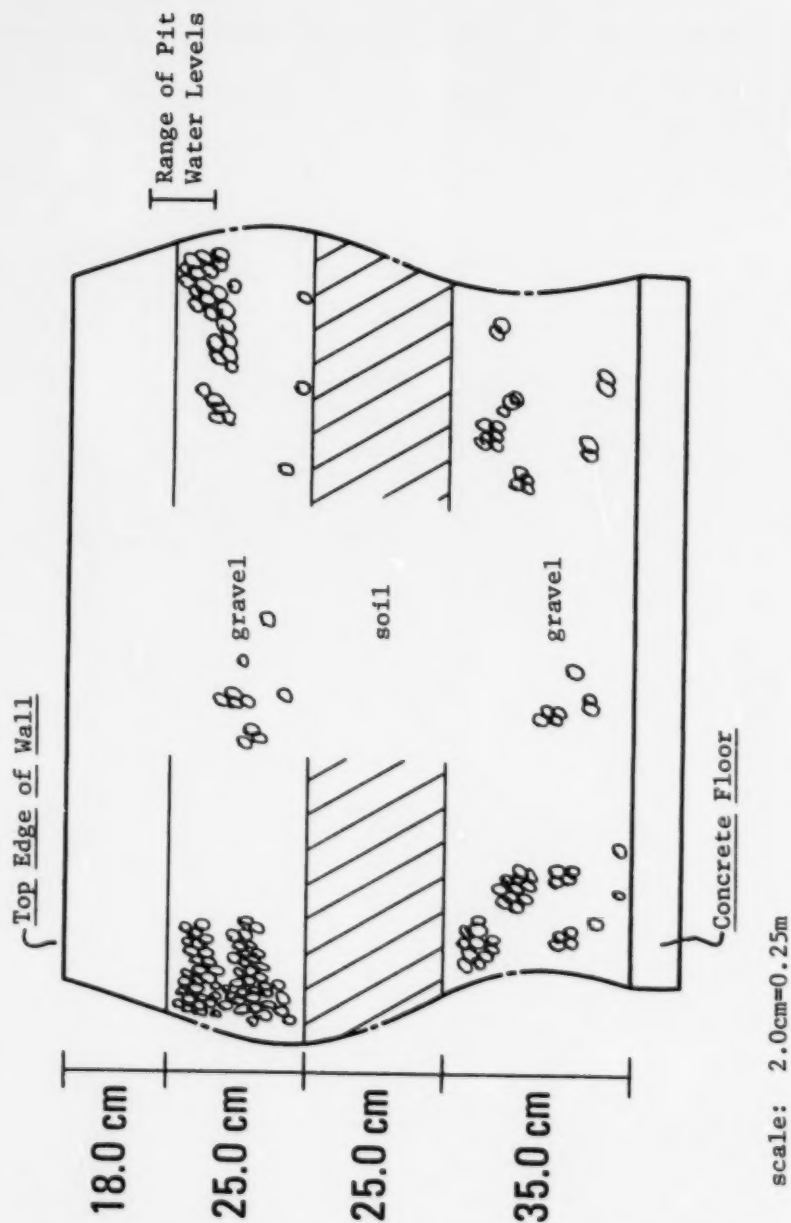


Figure 111. Internal view of Horticultural Research Station macropit.

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temperature, air humidity, water temperatures, wind movement, precipitation, pit evaporation, and standard class a pan evaporation. In addition, a measure of pit water level relative to the gravel surface was recorded (Figure 112). Data collected off-site were solar radiation (16.0 km south) and wind direction (72.0 km south). These off-site measurements should generally apply due to the very flat, uniform terrain of the region. Monthly means for all data are presented in Table 59.

SINGLE FACTOR ANALYSIS

Analysis of individual variables showed only relative pit water level (RPWL), pan evaporation (PANEVAP), and solar radiation (SRAD) to have appreciable predictive abilities toward pit evaporation (PITEVAP) from year to year and throughout the range of pit water levels. This consistency was not evident in air temperature, air humidity, or wind measurements, although one or more of these did prove to have predictive value dependent upon year and pit water level.

Relative Pit Water Level (RPWL)

RPWL ranged from 3.8 cm below to 4.8 cm above the gravel surface. Fluctuations within this range resulted in dramatic changes in the evaporative surface. At high levels, the surface was relatively plain and undisrupted while at low levels, it was completely covered by almost 4.0 cm of gravel. This drastic change in evaporative surface proved to have an appreciable effect on pit evaporation.

A comparison of RPWL Models for 1978, 1979 and Overall (all data combined) shows little discrepancy in evaporation response (Figure 113). The cubic response probably results from two phenomena; the submergence of the evaporative surface below a relatively impervious gravel layer at RPWL values less than 10.0 cm, and the creation of a very shallow depth of water emerging through and covering the gravel surface at RPWL values between 10.0 and 17.0 cm. The first phenomena results in a gravel mulch which acts to protect the water surface from climatological factors and which, thereby, becomes a major influence on evaporation rate. The second phenomena allows for direct solar and conductive heating of a shallow depth of water. The resulting rise in water surface temperatures and greater surface exposure are probably responsible for the marked increase in evaporation at RPWL values from 10.0 to 15.0 cm. As relative water depth advances beyond 15.0 cm, this heating effect diminishes and the evaporation rate subsides.

RPWL exhibits another peculiarity as it relates to pit evaporation, that is, a greater variability in evaporation at pit water levels above 12.7 cm. This increased variability indicates that evaporation rate begins to respond more to climatic factors as the water surface emerges from beneath the gravel. For this reason, the overall data was divided into low and high RPWL for statistical analysis (Figure 114). At high water levels (RPWL >12.7 cm) no appreciable relationship was found. However, a significant predictive

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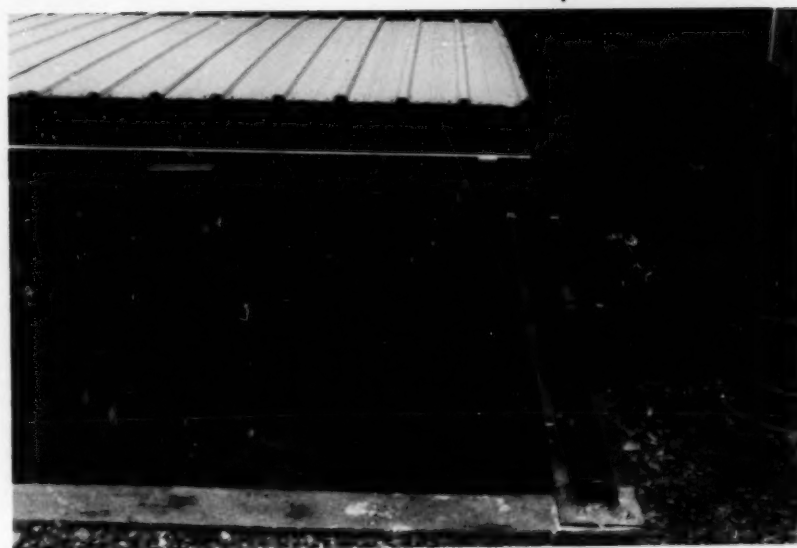


Figure 112. Horticultural research station disposal facility area. Top photo: layout of facility showing weather station in foreground with macropit and adjacent storage building upper right. Bottom photo: close-up of macropit showing mobile cover and track system. Note exposed water surface (high water level).

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TABLE 59. MONTHLY MEANS FOR EVAPORATION & CLIMATOLOGICAL DATA AT HORTICULTURAL RESEARCH STATION.
1978 & 1979

Measurement	Units	May*	June+	July+	Aug+	Sept+	Oct*	Nov*
Relative Pit Water Level (RPWL)	cm	12.19	13.07	14.30	14.90	11.44	9.136	7.838
Solar Radiation (SRAD)	ly	520.8	564.4	552.0	477.1	452.8	261.7	260.3
Pit Evaporation (PITEVAP)	1/m ²	2.492	4.400	3.962	3.947	2.281	0.837	0.393
Pan Evaporation (PANEVAP)	1/m ²	5.250	7.405	5.788	5.039	5.308	2.729	2.653
Pit Temperature Minimum (PITTMIN)	°C	14.33	15.91	19.73	18.65	17.50	7.941	6.800
Pit Temperature Maximum (PITTMAX)	°C	18.66	20.33	23.19	22.45	21.50	10.94	9.600
Pan Temperature Minimum (PANTMIN)	°C	11.26	15.00	17.96	16.08	13.77	3.470	2.400
Pan Temperature Maximum (PANTMAX)	°C	23.53	29.13	31.61	29.88	28.83	14.76	13.80
Air Temperature Minimum (AIRTMIN)	°C	11.00	13.69	16.55	15.82	13.08	0.705	0.600
Air Temperature Maximum (AIRTMAX)	°C	25.85	26.52	27.31	27.10	27.04	15.11	15.80
Humidity Minimum (HUMMIN)	%	38.75	42.71	52.73	52.19	43.29	38.94	36.60
Humidity Maximum (HUMMAX)	%	87.75	90.45	96.89	97.38	97.79	95.29	92.20
Wet-Bulb Depression Minimum (WBDMIN)	°C	0.750	1.150	0.507	0.653	0.625	0.276	0.440
Wet-Bulb Depression Maximum (WBDMAX)	°C	6.600	8.069	6.728	6.529	7.375	6.752	7.160
Wind Movement (PANWIND)	km	14.94	16.50	10.08	11.32	12.00	15.71	16.77
Precipitation (PRECIP)	mm	1.083	4.059	3.938	3.335	1.133	0.178	0.033

* Data from 1978.

+ Data from 1978 and 1979.

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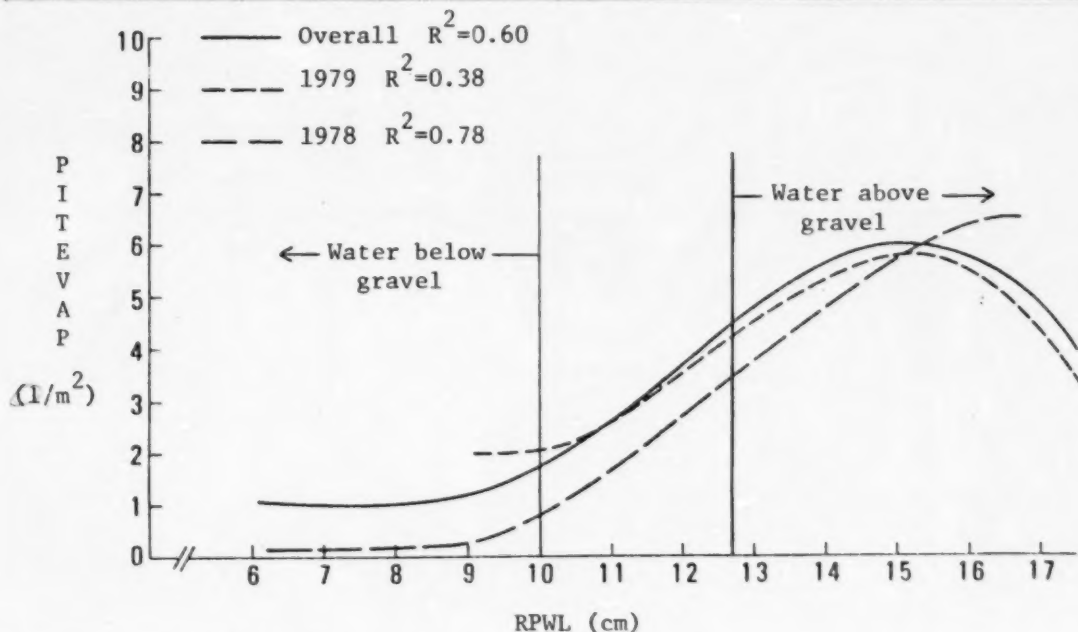


Figure 113. Relationship of pit evaporation (PITEVAP) to relative pit water level (RPWL) during 1978, 1979, and overall (all data combined).

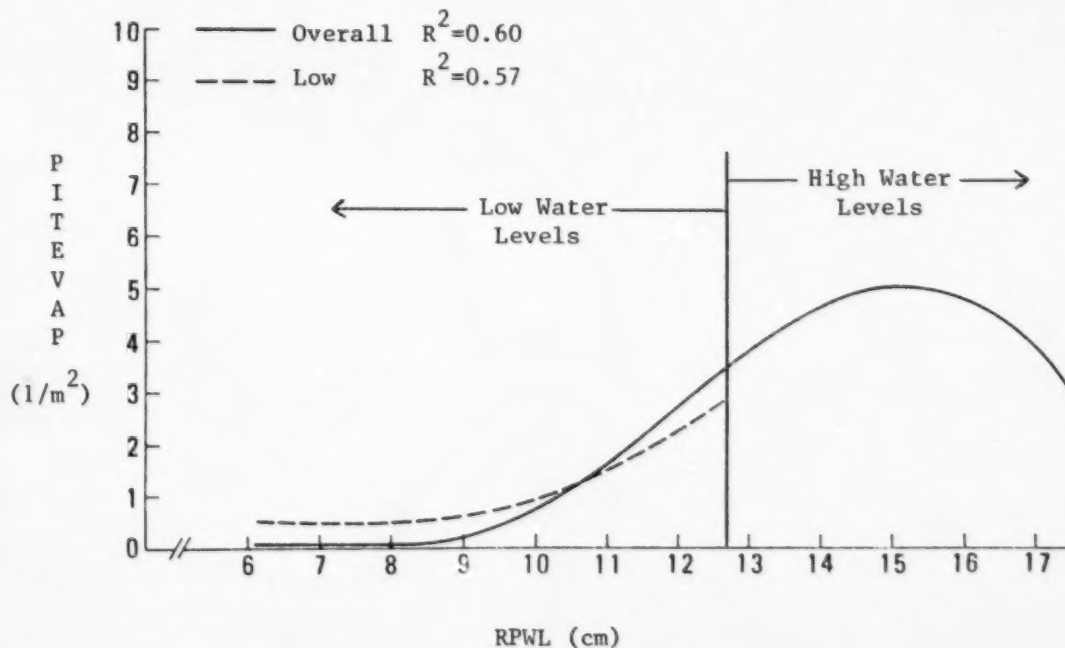


Figure 114. Relationship of pit evaporation (PITEVAP) to relative pit water level (RPWL) at low pit water levels and overall.

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ability was exhibited by RPWL at low pit water levels. Thus, there appear to be two distinct situations under study; pit evaporation at high water levels and pit evaporation at low water levels. *under study*
2 words

These results allow some insight into the better fit of RPWL in 1978 compared to 1979 ($R^2 = 0.78$ and 0.38 respectively). The fact that much of the 1978 data were collected at water levels lower than those experienced in 1979 and conversely that some of the 1979 data were collected at levels higher than those experienced in 1978 might explain some of the discrepancy in fit between the two years.

Solar Radiation (SRAD)

SRAD provides the energy for evaporation and could be a good predictor of pit water loss especially at high water levels. Comparison of models for 1978, 1979, and Overall shows a linear response, but again 1978 exhibits a much better fit (Figure 115). In addition, pit evaporation responded more to increases in solar radiation during 1978 than during 1979. This lack of homogeneity in response and fit is not readily explainable except to say that several non-quantifiable factors might be involved. Two such factors are the yearly differences in volume, frequency, and type of chemicals disposed, and the fact that data were collected from May through mid-November during 1978 versus June through mid-September during 1979. However, the poorer fit of 1979 data could be unique to the 1979 season. Evidence for this is seen in the similar lack of consistent correlation between solar radiation and pan evaporation for the two years (1978 $r = 0.83$, 1979 $r = 0.53$). Response to solar radiation at low versus high pit water levels is congruent with the assumed protective nature of the gravel surface, the response being much greater at high levels (Figure 116). Thus, solar radiation showed some predictive ability both from year to year and at high and low pit water levels.

Pan Evaporation (PANEVAP)

Measurements of evaporation from a standard class A pan represent an integration of the climatological factors affecting evaporation from a plane water surface. The application of this measurement to prediction of pit evaporation did not, however, show excellent results. Like solar radiation, pan evaporation is unable to account for a major portion of the variability in the data. However, it is more uniform in terms of fit from year to year and at high and low pit water levels (Figures 117 & 118).

The unique quality of the pan as an integrator of other factors may be responsible for this consistency. The predictive ability of pan evaporation is enhanced somewhat when the data are divided by water level (Figure 118). This increase in fit for both the high and low models over the 1978, 1979, and overall models lends support for the assumption that evaporation at high and low water levels is two distinct processes.

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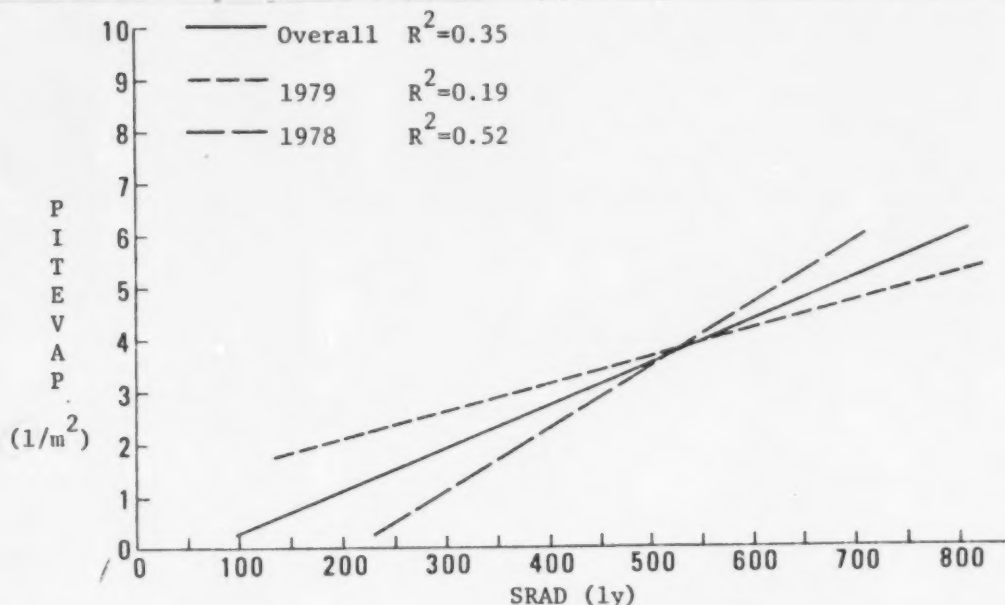


Figure 115. Relationship of pit evaporation (PITEVAP) to solar radiation (SRAD) during 1978, 1979, and overall (all data combined).

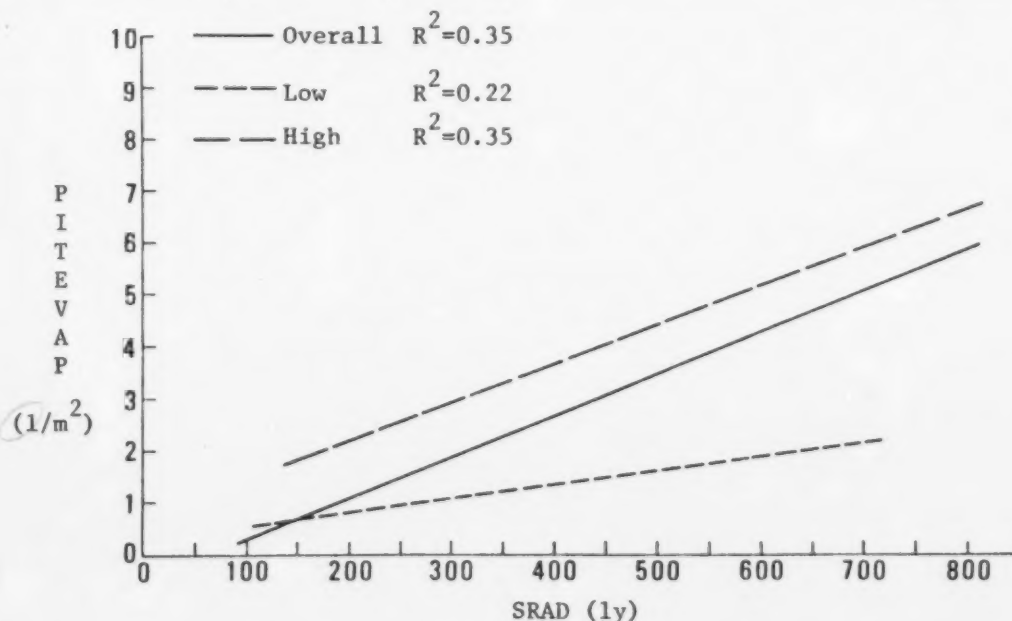


Figure 116. Relationship of pit evaporation (PITEVAP) to solar radiation (SRAD) at low and high pit water levels and overall.

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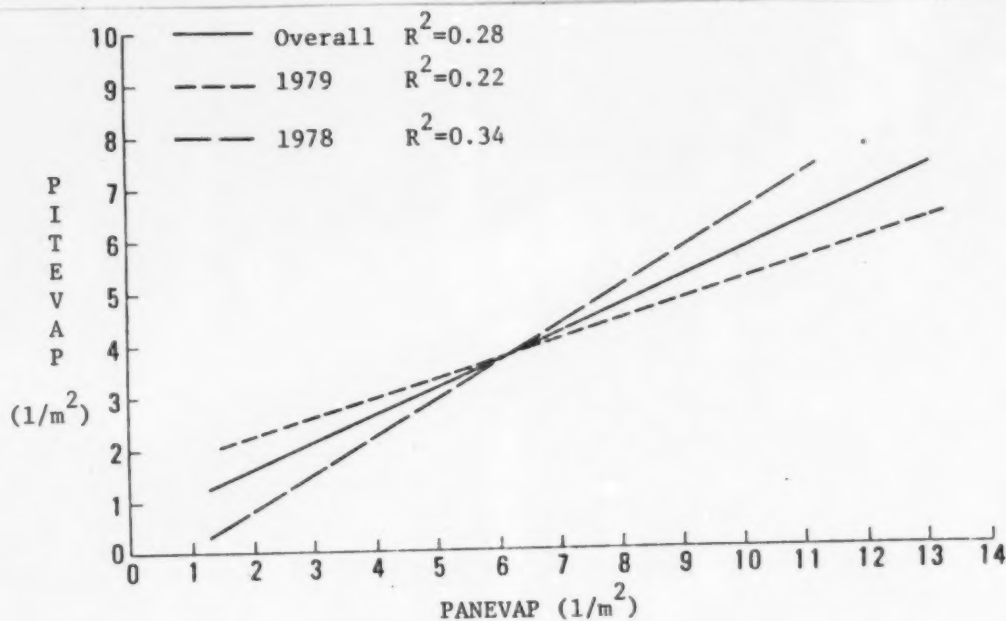


Figure 117. Relationship of pit evaporation (PITEVAP) to pan evaporation (PANEVAP) during 1978, 1979, and overall (all data combined).

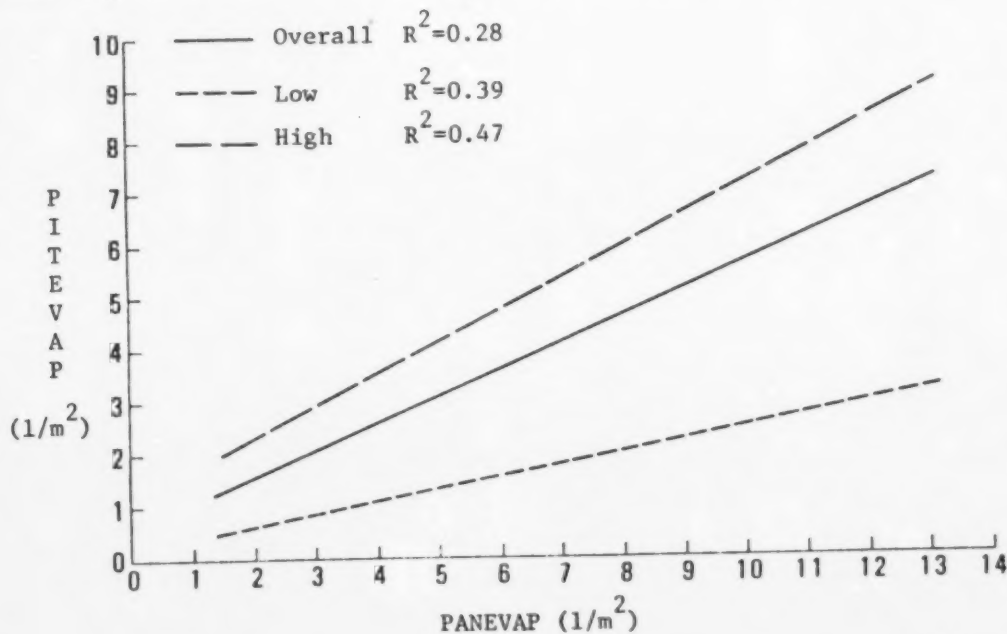


Figure 118. Relationship of pit evaporation (PITEVAP) to pan evaporation (PANEVAP) at low and high pit water levels and overall.

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Relative pit water level, solar radiation, and pan evaporation have each shown some consistent predictive ability toward pit evaporation. However, no relationship between pit water level and pit evaporation exists at high water levels. In addition, the relationships of pit evaporation to solar radiation and to pan evaporation are largely dependent upon pit water level. These facts indicate strongly that the gravel surface separates pit evaporation into two distinct classifications--evaporation at high water levels and evaporation at low water levels.

MULTIPLE FACTOR ANALYSIS

Multiple regression analyses were conducted for 1978, 1979, high water levels and low water levels. The models developed apply strictly to the year or pit water level range from which each was derived. Each model defines that set of factors which best predicts pit evaporation (PITEVAP) under the operational and climatological conditions experienced.

MODEL ONE: 1978 DATA ($R^2 = 0.84$)

PITEVAP = - 5.49 + 0.78 RPWL - 0.04 HUMMIN + 0.66 + [- 1.71 (E), - 1.14 (SE), - 1.00 (S), - 0.60 (SW), + 0.19 (W), + 0.64 (NW), + 0.00 (N)] * + 0.05 PANWIND.

Model One was developed using all available data for 1978. The large positive contribution of pit water level is consistent with the relationship defined in single factor analysis. Wind direction (WDIR), wind movement (PANWIND), and minimum relative humidity (HUMMIN) are present, but their contributions are slight by comparison. Winds from seven of eight possible compass directions exert various effects on pit evaporation. Winds from the east, southeast, south, and southwest have a negative influence whereas westerly and northwesterly winds have more of a drying effect relative to the north wind which was used as a zero reference. This is generally consistent with the expected moisture content of these winds and with the obstruction pattern around the pit. Wind movement shows the expected positive effect which is modified by wind direction and relative humidity. The negative effect of minimum relative humidity is appropriate to its inverse relationship to evaporation rate.

* Wind direction (WDIR) from compass directions: East (E), Southeast (SE), South (S), etc.

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MODEL TWO: 1979 DATA ($R^2 = 0.73$)

PITEVAP = - 5.39 + 0.44 RPWL + 0.003 SRAD + 0.58 PANEVAP - 1.08 WBDMIN
- 0.29 WBDMAX + 0.05 PANWIND - 0.10 PRECIP.

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Model Two was developed from all available data for 1979. Pit water level is a major influence once again but is reduced from its strength in 1978. The presence of solar radiation and pan evaporation in the model completes the three variables expected to contribute based on single factor analysis. Wind movement maintains its influence but wind direction is lost while precipitation (PRECIP) contributes a negative effect. The negative effects of wet-bulb depression minimum and maximum (WBDMIN, WBDMAX) are interpreted as having correctional value since their normal relationship to evaporation is positive.† The reduced influence of pit water level and greater contribution of climatological factors probably is a reflection of the higher water levels experienced during 1979.

MODEL THREE: LOW DATA ($R^2 = 0.79$)

PITEVAP = - 5.22 + 0.37 RPWL + 0.25 PANEVAP - 0.06 AIRTMAX + 0.03
HUMMAX + 0.39 + [- 0.91 (E), + 0.31 (SE), - 0.33 (S), - 0.53 (SW), + 0.21
(W), - 0.09 (NW), + 0.00 (N)]*.

Model Three was developed from all data collected at low pit water levels during both 1978 and 1979. Pit water level and pan evaporation are highly significant positive influence in the model. Maximum air temperature (AIRTMAX) and maximum relative humidity (HUMMAX) while highly significant exert influences opposite of that expected. Once again, this sign reversal is attributed to the assumed corrective nature of these variables in this particular model. Wind direction exhibits the same general influence as in the 1978 model. The deletion of solar radiation is expected in view of its rather high correlation with pan evaporation ($r = 0.74$).

The good fit of this model ($R^2 = 0.79$) and the non-excessive contribution of pit water level indicates that while the pit may respond differently to climatological factors at low water levels, it is certainly not completely isolated from them. This would suggest the possibility of predicting pit evaporation solely from climatological factors.

† It should be noted that the presence of variables which exerts effects opposite of their real physical relationship to evaporation might reduce the probability that the model will apply to areas of divergent climate.

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MODEL FOUR: HIGH DATA ($R^2 = 0.71$)

PITEVAP = - 3.35 + 0.28 RPWL + 0.0004 SRAD + 0.40 PANEVAP - 0.17

AIRTMIN + 0.08 AIRTMAX + 0.48 WBDMIN - 0.18 WBDMAX + 0.07 PANWIND

- 0.06 PRECIP.

Model Four, derived from all data collected at high pit water levels, demonstrates the reduced influence of pit water level once a plane evaporative surface is achieved. The presence of numerous climatological variables indicates the increasing importance of these factors as pit water level rises. Again, the possibility of predicting pit evaporation solely from climatological data becomes evident.

The four models discussed above describe the evaporative characteristics of this facility at this location. These models may not be applicable to similar facilities at other sites since wind direction and relative pit water level are used. The site specific nature of wind direction results from the numerous buildings and trees surrounding the pit while relative pit water level is specific to the disposal volume generated by the research station. Thus while the models are an aid to understanding the general predictability of pit evaporation, they may not be accurate in predicting evaporation at other locations. For this reason two additional models have been developed which do not utilize wind direction or pit water level.

MODEL FIVE: LOW DATA w/o WDIR or RPWL ($R^2 = 0.58$)

PITEVAP = 1.17 + 0.46 PANEVAP - 0.46 WBDMIN - 0.18 WBDMAX - 0.02

PANWIND - 0.04 PRECIP.

Model Five attempts to predict pit evaporation at low water levels without the aid of pit water level or wind direction. The model should apply to facilities which for safety reasons cannot have an exposed water surface. The fit of this model is much reduced from that obtained for the low model ($R^2 = 0.58$ vs. 0.79). Even so, its greater applicability to other locations should be clear. The model depends on pan evaporation as the one positive contributor. The sign reversals of wet-bulb depression minimum and maximum and wind movement probably reflect the fact that without an exposed water surface the pit cannot respond to these factors as efficiently as the pan.

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MODEL SIX: HIGH DATA w/o WDIR or RPWL ($R^2 = 0.67$)

PITEVAP = $1.10 + 0.003 \text{ SRAD} + 0.40 \text{ PANEVAP} - 0.15 \text{ AIRTMIN} + 0.08$

AIRTMAX + $0.56 \text{ WBDMIN} - 0.20 \text{ WBDMAX} + 0.06 \text{ PANWIND} - 0.07 \text{ PRECIP.}$

Model Six attempts to predict pit evaporation at high water levels without the aid of pit water level or wind direction. Its predictive ability compares favorably to that of the high model ($R^2 = 0.67$ vs. 0.71) and indicates the dispensability of relative pit water level. The model should predict quite well the daily evaporation from this type of pit at various other locations. The fact that it was developed from data collected at high water levels makes it a good tool for predicting the maximum evaporation and disposal capacity of the facility.

SUMMARY AND CONCLUSIONS

Each of the first four models discussed exhibits good predictive ability toward pit evaporation. Each is probably somewhat limited, however, to predicting evaporation at this site. The elimination of two key site-specific variables results in models which do not suffer this constraint but which do suffer some loss in predictive ability. These models, without wind direction and relative pit water level, depend entirely upon climatic factors and, therefore, should be applicable to other parts of the central U.S. and possibly beyond. However, any of the site-specific models might also have this application if the probable operational water level and the prevailing wind direction are determined and used in the model. The choice of which model or models to use in determining pit size will ultimately depend on the availability of data and an evaluation of the operational situation and the climatological exposure of the site. Certainly more than one model should be used if possible. Further, the values generated from any model should be viewed as an estimate of the maximum possible evaporation rate.

Results from this study indicate that multiple linear regression models can predict pit evaporation from climatological and pit related data. These predictions could in turn be used to determine the minimum pit surface area required to accommodate a particular volume of continuous disposal. A sample calculation of such a prediction is given in Table 60.

† The obstruction pattern surrounding the disposal site should be evaluated prior to determining which wind direction might be appropriate. The pit under study is heavily protected on the East and South which probably accounts for some part of the negative contributions of winds from these directions. Certainly an unobstructed easterly or southerly wind might have a more positive effect than the models indicate.

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TABLE 60. SAMPLE DETERMINATION OF PIT SURFACE AREA REQUIRED TO
ACCOMMODATE A PARTICULAR VOLUME OF CONTINUOUS DISPOSAL

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DATA: From Table 59, monthly means for July. Assume a westerly prevailing
wind with open exposure and mean relative pit water level of 15.5 cm
(approximately 3.75 cm above gravel surface).

MODEL ONE: $\text{PITEVAP} = - 5.49 + 0.78 \text{ RPWL} - 0.04 \text{ HUMMIN} + 0.66$
 $+ [- 1.71 \text{ (E)}, - 1.14 \text{ (SE)}, -1.00 \text{ (S)}, - 0.60 \text{ (SW)}, +$
 $0.19 \text{ (W)}, + 0.64 \text{ (NW)}, + 0.00 \text{ (N)}]^1 + 0.05 \text{ PANWIND}.$

COMPUTATION: $\text{PITEVAP} = - 5.49 + 0.78 (15.5) - 0.04 (52.7) +$
 $0.66 + 0.19 + 0.05 (10.1).$

SOLUTION: $\text{PITEVAP} = 5.85$ liters/square meter of surface. To accomodate
average daily disposal of 200 liters during July, a surface area
of 34.2 square meters (200 liters $\div$ 5.85 liters per square meter)
is required.

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